Docket No.: 2927-0152P

REMARKS

The Examiner is thanked for the thorough examination of the application. No new matter is believed to be added to the application by this Amendment.

Entry Of Amendment

Entry of this Amendment under 37 C.F.R. §1.116 is respectfully requested because it places the application in condition for allowance. Alternately, entry is requested as placing the application in better form for appeal.

Status Of The Claims

Upon entry of this Amendment, claims 1-19 are pending in the application. Claim 3 has been amended to improve the language without reducing the scope. Claim 8 has been amended to clarify the language, and the clarification is supported at page 13, lines 8-9 of the substitute specification. Claim 18 is supported by claims 1, 2 and 3. Claim 19 is supported by claims 1, 2 and 5.

Rejections 35 U.S.C. §112 (Paragraphs 1-5 of the Office Action)

Claims 2, 6 and 8 are rejected under 35 U.S.C. §112, first paragraph as not being enabled or complying with the written description requirement. Claims 3-6, 8 and 9 are rejected under 35 U.S.C. §112, second paragraph as being indefinite. Applicants traverse.

In paragraph 1 of the Office Action, the Examiner asserts that the claim 6 ("not 8 ADM/REG/imb

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less than 0.5 mol% of said organometallic salt is single-ionized") is not adequately

described in the specification. Support for this limitation is found at page 11, lines 15-16

of the specification ("not less than 0.5 % of said organometallic salt is single-ionized").

Since this is a property intrinsic to the organometallic salt and not compared to another

component, using mol% would be assumed by one of ordinary skill.

Alternately, mol% can be found without undue experimentation. The test of

enablement is whether one reasonably skilled in the art could make or use the invention

from the disclosures in the patent coupled with information known in the art without

undue experimentation." United States v. Telectronics, Inc., 8 USPQ2d 1217 (Fed. Cir.

1988); In re Stephens, 188 USPQ 659 (CCPA 1976). "A patent may be enabling even

though some experimentation is necessary; the amount of experimentation, however,

must not be unduly extensive." Utter v. Hiraga, 6 USPQ2d 1709, 1714 (Fed. Cir. 1988).

In paragraphs 1 and 5 of the Office Action, the Examiner finds the term "main

component" to be inadequately defined. However, claim 8 has been amended to set

forth this quantity as being not less than 50 wt%.

In paragraphs 2-4 of the Office Action, the Examiner asserts that there is

inadequate written description and inadequate enablement of JIS K6262, JIS K6911

and JIS K-6253 recited in claim 2. However, MPEP 2163 points out that to satisfy the

written description requirement, a patent specification must describe the claimed

invention in sufficient detail that one skilled in the art can reasonably conclude that the

inventor had possession of the claimed invention. See, e.g., Moba, B.V. v. Diamond

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Automation, Inc., 325 F.3d 1306, 1319, 66 USPQ2d 1429, 1438 (Fed. Cir. 2003); Vas-

Cath, Inc. v. Mahurkar, 935 F.2d at 1563, 19 USPQ2d at 1116. Also, there is no

requirement in the MPEP that all methodologies be in the English language or that the

person of ordinary skill is necessarily restricted to using the English language.

Further, these Japanese industrial standards are well known and would be

known as such by one having ordinary skill. Also, MPEP 2164.01 points out that the

standard for determining whether the specification meets the enablement requirement

was cast in the Supreme Court decision of Mineral Separation v. Hyde, 242 U.S. 261,

270 (1916) which posed the question: is the experimentation needed to practice the

invention undue or unreasonable? That standard is still the one to be applied. In re

Wands, 858 F.2d 731, 737, 8 USPQ2d 1400, 1404 (Fed. Cir. 1988).

However, in this case no experimentation is required, and one of ordinary skill

need only to obtain a translation of JIS K6262, JIS K6911 and JIS K-6253. For the

Examiner's convenience, please find attached English translations of JIS K6262, JIS

K6911 and JIS K-6253.

In paragraph 5 of the Office Action, the Examiner finds claim 3 to have

insufficient antecedent basis. However, claim 3 has been amended to be clear definite

and have full antecedent basis.

These rejections are overcome and withdrawal thereof is respectfully requested.

Rejections under 35 U.S.C. §103 (a) (Paragraphs 7-11 of the Office Action)

Claims 1-10 and 12-17, are rejected under 35 U.S.C. §103(a) as being obvious

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over Vreeland '001 or '457 (U.S. Patent 5,541,001; U.S. Patent 5,571,457) or Gloyer

(U.S. 2001/0046576) each in view of Barksby (U.S. Patent 6,420,445) and Knobel (U.S.

Patent 5,110,669). The Examiner adds the teachings of Nogami (U.S. Patent

5,618,646) or Priebe (U.S. Patent 5,869,188) to the foresaid rejection to object claim 11.

Applicants traverse.

The Present Invention And Its Advantages

The present invention pertains to a conductive urethane composition and a

conductive roller composed of the conductive urethane composition. The present

invention improves the mixing of the urethane composition that can be effectively used

for conductive rollers such as a charging roller, a developing roller, a toner supply roller,

or a transfer roller of a copying machine or a printer.

One of the many novel features of the present invention's conductive urethane

composition rollers resides in a low electric resistance of less than $10^{9.0} \Omega \cdot \text{cm}$ (claim 2)

or less than $10^{8.0}~\Omega$ ·cm (claims 3, 18 and 19). In the invention, the average value of

non-saturation of the constituent polyether polyol is set to not more than 0.025

milliequivalents/g (claims 1, 18 and 19).

Distinctions Of The Invention Over The Applied Art

All of the cited art fails to disclose or suggest the 0.025 milliequivalent/g limit of

non-saturation recited in claim 1 of the present invention.

Vreeland '001 pertains to a polyurethane suitable for a roll that is formed by

reacting a polyisocyanate polymer, a polyol and a conductivity control agent that is an

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ammonium tetrahaloferrate salt.

Vreeland '457 pertains to the polymeric material formed from a polyurethane

obtained by reacting a polyisocyanate polymer with a polyol blended with a ferric halide

complex.

Glover pertains to a polyurethane elastomer formed from a polyisocyanate

prepolymer and a polyether polyol prepolymer (claim 1). Glover, in claims 2 and 3.

recites polyol charge-control agents.

Vreeland '001, Vreeland '457 and Glover all fail to disclose a degree of

unsaturation of the polyether polyol that is not more than 0.025 milliequivalents/g (claim

1 of the present invention). The Examiner unequivocally admits to this failure of the

primary references in paragraph 8 of the Office Action.

Also, the Vreeland patents use iron compounds as a charge control agent.

Gloyer uses a conductive polyol as the charge control agent. In contrast, the present

invention preferably uses organic ionic-conductive agents that are organometallic salts

which preferably have fluoro groups and/or sulfonyl groups (claims 4, 5 and 19). The

preferred examples of organometallic salts are found at page 11. lines 12-14 of the

substitute specification (claim 17).

At paragraph 8 of the Office Action, the Examiner unequivocally admits that the

primary references fail to specifically recited the present invention's claimed polyether

polyol having the claimed degree of unsaturation.

The Examiner then turns to Barksby for teachings pertaining to unsaturation.

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Barksby column 7, line 44 mentions "low unsaturation polyoxypropylene glycol."

However, Barksby fails to disclose the 0.025 milliequivalent/g limit of non-saturation

recited claim 1 of the present invention.

The Examiner refers to columns 7 and 8 of Knobel for teachings pertaining to

ionizable salts. However, although Knobel may refer to group IA (column 7, line 28),

Knobel fails to specifically disclose the specific lithium salts cited at page 11, lines 12-16

of the substitute specification and set forth in claim 17.

The Examiner turns to Nogami for teachings pertaining to the treatment of the

metal shaft to reject claim 11. However, these teachings of Nogami fail to address the

deficiencies of the other cited art references in suggesting a claimed embodiment of the

present invention.

That is, all of the cited art utilized by the Examiner fails to teach or suggest "an

average value of a non-saturation degree of said polyether polyol is set to not more than

0.025 milliequivalents/g" such as is set forth in claims 1, 18 and 19. As a result, the

cited prior art fails to teach or suggest each and every element of the independent

claims of the present invention. To establish prima facie obviousness of a claimed

invention, all the claim limitations must be taught or suggested by the prior art. In re

Royka, 490 F.2d 981, 180 USPQ 580 (CCPA 1974). "All the words in a claim must be

considered in judging the patentability of that claim against the prior art." In re Wilson,

424 F.2d 1382, 1385, 165 USPQ 494, 496 (CCPA 1970).

Therefore, one having ordinary skill in the art would not be motivated by Vreeland

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'001 or '457, Gloyer, Barksby, Knobel, Nogami and Priebe to produce a claimed

embodiment of the present invention. A prima facie case of obviousness has thus not

been made.

Further, even if one assumes arguendo that the cited art is sufficient to allege

prima facie obviousness, this obviousness would be fully rebutted by the unexpected

results shown in the Examples of the present invention for such properties as low

staining, picture quality and uniformity of electrical resistance. The advantages of the

invention are thus clear.

These rejections are overcome and withdrawal thereof is respectfully requested.

Information Disclosure Statement

The Examiner is thanked for considering the Information Disclosure Statement

filed July 30, 2003 and for making the initialed PTO-1449 form of record in the

application in the Office Action mailed March 22, 2005.

Prior Art

The prior art cited but not utilized by the Examiner indicates the status of the

conventional art that the invention supercedes. Additional remarks are accordingly not

necessary.

The Drawings

The Examiner has accepted the drawings in the Office Action mailed December

2, 2005.

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Foreign Priority

The Examiner has acknowledged foreign priority and noted that a certified copy

of the priority document has been received in the Office Action mailed March 22, 2005

and in the Office Action mailed December 2, 2005.

<u>Assignment</u>

The Assignment was recorded on July 30, 2003 at reel 014362, frames 0265-

0267.

Conclusion

The Examiner's rejections have been overcome, obviated or rendered moot. No

issues remain. The Examiner is accordingly respectfully requested to place the

application in condition for allowance and to issue a Notice of Allowability.

Should there be any outstanding matters that need to be resolved in the present

application, the Examiner is respectfully requested to contact Robert E. Goozner, Ph.D.

(Reg. No.42,593) at the telephone number of the undersigned below, to conduct an

interview in an effort to expedite prosecution in connection with the present application.

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If necessary, the Commissioner is hereby authorized in this, concurrent, and future replies, to charge payment or credit any overpayment to Deposit Account No. 02-

2448 for any additional fees required under 37 C.F.R. § 1.16 or under 37 C.F.R. § 1.17;

particularly, extension of time fees.

Dated: March 2, 2006

Respectfully submitted,

Andrew D. Meikle

Registration No.: 32,868

BIRCH, STEWART, KOLASCH & BIRCH, LLP

Docket No.: 2927-0152P

#42,593

8110 Gatehouse Road

Suite 100 East P.O. Box 747

Falls Church, Virginia 22040-0747

(703) 205-8000

Attorney for Applicant

ADM/REG/jmb

Attachments: JIS K 6911 – 1995

JIS K 6253 – 1997

JIS K 6262 – 1997

JAPANESE INDUSTRIAL STANDARD

Testing methods for thermosetting plastics

JIS K 6911-1995

Translated and Published

by.

Japanese Standards Association

Printed in Japan

In the event of any doubt arising, the original Standard in Japanese is to be final authority

Errata for JIS (English edition) are printed in *Standardization Journal*, published monthly by the Japanese Standards Association.

Errata will be provided upon request, please contact: Business Department,
Japanese Standards Association
4-1-24, Akasaka, Minato-ku,
Tokyo, JAPAN 107
TEL. 03-3583-8002
FAX. 03-3583-0462

Errata are also provided to subscribers of JIS (English edition) in Monthly Information.

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JAPANESE INDUSTRIAL STANDARD

JIS

Testing methods for thermosetting plastics

K 6911-1995

- 1. <u>Scope</u> This Japanese Industrial Standard specifies the general methods of testing thermosetting plastics.
 - Remarks 1. The following standards are cited in this Standard.

JIS B 1352 Taper pins

JIS B 4410 Taper pin reamers

JIS B 7502 Micrometer callipers

JIS B 7503 Dial gauges

JIS B 7507 Vernier, dial and digital callipers

JIS B 7512 Steel tape measures

JIS B 7513 Precision surface plates

JIS B 7514 Steel straightedges

JIS B 7516 Metal rules

JIS B 7601 Trip balances

JIS B 7726 Rockwell and Rockwell superficial hardness testing machines

JIS C 1102 Electrical indicating instruments

JIS C 1302 Insulation resistance testers

JIS C 2320 Electrical insulating oils

JIS G 4051 Carbon steels for machine structural use

JIS K 8034 Acetone

JIS K 8951 Sulfuric acid

JIS R 3503 Glass apparatus for chemical analysis

JIS R 6252 Abrasive papers

JIS Z 8101 Glossary of terms used in quality control

JIS Z 8401 Rules for rounding off of numerical values

JIS Z 8801 Test sieves

- 2. The units and numerical values given in { } in this Standard are in accordance with the traditional unit system and appended for informative reference.
- 2. <u>Definitions</u> For the purpose of this Standard, the definitions of principal terms are as follows:
- (1) apparent density The mass of moulding material per unit volume.
- (2) <u>bulk factor</u> The quotient of the density of moulding product divided by the apparent density of the moulding material used for the moulding product. The bulk factor is equal to the quotient of the volume of the moulding material required for moulding divided by the volume of the moulded product.

- (3) <u>free water content</u> The water to be lost from a moulding material when the material is dried at room temperature.
- (4) warping rate The concave or convex deformation of material in the direction parallel to the edge, represented by the maximum warp for a length of 1000 mm in percent.
- (5) <u>twisting rate</u> The concave or convex deformation of material in the diagonal direction, represented by the maximum twist of a length of 1000 mm in percent.
- (6) mould shrinkage coefficient Moulding material shrinks smaller than the mould dimension when it is taken away after moulded from a mould and allowed to cool down in the air. The difference between the mould dimension and the dimension of the moulding product is called "mould shrinkage". And the percentage of the mould shrinkage to the mould dimension is the mould shrinkage coefficient.
- (7) rate of shrinkage on heating The shrinkage on heating is the difference between the dimension of the moulding product which has been moulded from a moulding material in a mould, taken out of the mould, allowed to cool down in the air (the dimension of the product in this state is termed initial dimension), and that of the product further conditioned in a thermostatic oven maintained at a specified temperature, its rate is the ratio of the shrinkage to the initial dimension in percent.
- (8) withstand voltage The property of a material such that its test piece withstands without breakdown the specified voltage (the specified potential gradient × the thickness of test piece) applied for 1 min.
- (9) <u>flatwise withstand voltage</u> The withstand voltage of a laminate when the voltage is applied vertically to its laminae.
- (10) edgewise withstand voltage The withstand voltage of a laminate when the voltage is applied parallel to its laminae.
- (11) <u>dielectric breakdown strength</u> Under the specified test conditions, the quotient of the minimum effective voltage at the breakdown of the test piece (breakdown voltage) divided by the distance between the two electrodes (thickness of test piece).
- (12) insulation resistance The quotient of the d.c. voltage applied between two electrodes divided by the total current flowing between them. It includes both the volume resistance and surface resistance of the test piece. In the case of laminates, it indicates the edgewise insulation resistance when the voltage is applied parallel to laminae.
- volume resistance The quotient of the d.c. voltage applied between the two electrodes divided by the current through a unit volume of the test piece placed between the electrodes.
- (14) surface resistance The quotient of the d.c. voltage applied between two electrodes attached on one side of the test piece divided by the current flowing along the surface layer.
- (15) volume resistivity The quotient of the potential gradient parallel to the current within the test piece divided by the current density. This value equals the volume resistance between the two electrodes forming the two opposite sides of a cube with 1 cm edges.

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- (16) surface resistivity The quotient of the potential gradient parallel to the current along the surface of the test piece divided by the current per unit width of the surface. This value equals the surface resistance between the two electrodes forming the opposite two sides of a square with 1 cm sides.
- (17) dielectric constant The amount of electrostatic energy stored in a unit volume of a substance in a unit electric field. It is called "dielectric constant (ε) " and expressed as the quotient of equivalent parallel capacitance of a capacitor employing the test piece as the dielectric (C_x) divided by that of the same capacitor when air (under the standard condition the dielectric constant of air can be taken for that of vacuum) is used as the dielectric (C_0) , measured under a specified frequency.

i.e.,
$$\varepsilon = \frac{C_x}{C_0}$$

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(18) dielectric dissipation factor Dielectric dissipation factor is the tangent of complementary angle to a dielectric phase angle (tan δ). The dielectric phase angle is the phase difference angle (θ) between the sine-wave voltage applied to the test piece and the current component having the same frequency as the applied voltage caused by the voltage application.

Remarks: The cosine of the dielectric phase angle (θ) , i.e., dielectric power factor, is sometimes used in place of dielectric dissipation factor, since dielectric power factor can be regarded nearly equal to dielectric dissipation factor, if tan $\delta < 0.1$.

- (19) <u>arc resistance</u> The period of time for which the test piece can withstand the exposure to arc.
- (20) Rockwell hardness A number calculated by the following equation from the net increase in the depth of impression when the load on a steel ball indenter is increased from a fixed minor load to a major load and then returned to the minor load, by employing an appropriate ball indenter specified depending on the hardness of the test piece.

$$H_R = 130 - 500h$$

where, H_R : Rockwell hardness

h: net increase in the depth of impression (mm)

In the above procedure, use a steel ball of 6.350 mm diameter for the measurement of M scale, with the minor load of 98 N {10 kgf} and major load of 980 N {100 kgf}, and use a steel ball of 12.700 mm diameter for the measurement of R scale, with the minor load of 98 N {10 kgf} and major load of 590 N {60 kgf}.

(21) Barcol hardness The maximum reading of a Barcol impressor of Type 934-1, when the test piece is indented by applying a load on the indenter of the instrument.

- (22) <u>flexural strength</u> The maximum flexural stress in the test piece when it is supported at both ends as a both-end-supported beam and applied with a concentrated load on the upper face at the centre of span. However, for a laminated tube of not more than 100 mm inside diameter, the flexural strength is the maximum breaking stress in the test piece when it is placed between two parallel boards and uniformly loaded on the axis.
- (23) bending modulus of elasticity The resistivity of the test piece against deformation by flexural stress, in the straight portion of the load-strain curve within the elastic range. It is expressed in the flexural stress per unit strain.
- (24) tensile strength The quotient of the maximum tensile breaking load applied on the test piece divided by the minimum original cross-sectional area of the test piece, i.e., the maximum tensile stress.
- (25) compressive strength The quotient of the maximum compressive breaking load applied on the test piece divided by the compressed original cross-sectional area of the test piece, i.e., the maximum compressive stress.
- (26) Charpy impact strength The quotient of the total impact energy absorbed when the test piece is broken in a Charpy impact testing machine divided by the original cross-sectional area of the test piece under the notch.
- (27) <u>Izod impact strength</u> The quotient of the total impact energy absorbed when the test piece is broken in Izod impact testing machine divided by the original width of the test piece at the notch.
- (28) bonding strength The load at the break of the laminated sheet when the load is applied parallel to its laminae by using a steel ball.
- (29) <u>deflection temperature under load</u> The temperature of the heat-transfer medium when the deflection of the test piece at the centre of span reaches a fixed amount, when a bar-shape test piece is supported at two points, applied with a fixed load at the centre of span and is heated from outside at a constant speed.
- (30) <u>burning resistance</u> The grade of burning resistance and self-extinguishing property of plastics. It is expressed by the duration of burning after ignition and burning distance.
- (31) flaming The state of burning with flames.
- (32) glowing The state of red-heating without flames.
- (33) water absorption The percentage of the mass of test piece which has increased by immersion in water for 24 h to its mass before immersion.
- (34) boiling water absorption The percentage of the mass of test piece which has increased by immersion in boiling water for 1 h to its mass before immersion.
- (35) boiling water resistance The resistivity of the test piece with appearance against immersion in boiling water for 15 min.
- (36) chemical resistance The resistivity of test piece with mass and appearance against immersion in a testing solution maintained at a constant temperature for a specified period of time.

3. Method of sampling and making test pieces

- 3.1 <u>Method of sampling</u> In the case of testing the quality of product manufactured in lots (1), samples (2) shall be taken in accordance with the following, unless otherwise specified.
- (1) Moulding material Make random sampling on a lot whose quality can be regarded as uniform so that the sample will represent the quality of the whole lot. The quantity of the sample shall be about two times as necessary for the test and the sample shall be contained in a vessel which can be closed tight.
- (2) <u>Laminates</u> Sample at random one piece of product as the sample from a lot of laminated sheets, rods or tubes that can be regarded as of the same quality, shape and dimensions.
 - Notes (1) The term lot used here means, as defined in S6 (stratified sampling) of clause 3. (5) in JIS Z 8101, moulding material, laminated sheets, rods and tubes of the same quality produced at the same time under the same conditions.
 - (2) The term sample used here means, as defined in S9 (cluster sampling) of clause 3. (5) in JIS Z 8101, one sampled from the same lot to be subjected to measurements and tests.

3.2 Method of making test pieces

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- (1) Moulding material When making each test piece by moulding the sample, use the metal mould which meets the requirements for the respective test piece, and mould under the conditions as appropriate to the sample.
- (2) <u>Laminated sheets</u> Unless otherwise specified, cut the test pieces from the ends of the sample lengthwise or crosswise, with care not to produce heat by machining.
- (3) <u>Laminated rods</u> Unless otherwise specified, cut the test pieces from the ends of the sample, with care not to produce heat by machining.
- (4) <u>Laminated tubes</u> Unless otherwise specified, cut the test pieces from the ends of the sample, with care not to produce heat by machining.

Remarks: Unless otherwise specified, the tolerances on the dimensions of test piece shall be \pm 5 % of the specified dimensions.

4. <u>Test items</u> The test items for each type of thermosetting plastic product shall be as given in Table 1.

Table 1. Test items

cable Mould-Lami-Lami-Lami-
sheet
<u>.</u>
2
2

Table 1. (continued)

Expres-	sion of test results				Occur-	rence of frac- ture				Mean	value			Mean		
Measured	value, unit or method of expres- sion					<u> </u>			MV/m	MV/m	MV/m			MD		
	Preconditioning of test piece			C-90 +4 b/90 + 9:0//6E	± 5) % RH		$0-0.5 \text{ h/90} \pm 2^{\circ}\text{C}$	$C_{-90}^{+4} h/20 \pm 2^{\circ}C/(65 \pm 5) \% RH$	C-90 ⁺⁴ h/20 ± 2°C/(65 + 5) % RH	D-48 ± 2 h/50 ± 3°C	$C_{-90}^{+4}_{-2}h/20 \pm 2^{\circ}C/(65 \pm 5) \% RH$	$D-48 \pm 2 \text{ h/50} \pm 3^{\circ}\text{C}$	$C-90^{+4}_{-2}h/20 \pm 2^{\circ}C/(65)$ $\pm 5) \% RH$	D-2 h/100°C + D-0.5 h/ 20 ± 10°C	$C_{-90}^{+4} h/20 \pm 2 \cdot C/(65 \pm 5) \% RH$	$D-2 \text{ h/100}^{\circ}\text{C} + D-0.5 \text{ h/}$ $20 \pm 10^{\circ}\text{C}$
	Size of test piece mm	Circular disc ϕ (60 to 100) × (2 ± 0.15)	Full dia. \times (2 ± 0.15)	Square plate ☐ (70 to 100) × (2 + 0.15)	Machined tube as Fig. 10, 80	Machined piece as Fig. 11, 70 square × (2 ± 0.15)	With electrode hole as Fig. 12, $75 \times 50 \times (3 \text{ to } 10)$	With electrode hole as Fig. 13, 40 × 20 × full thickness		Circular disc, ϕ (80 to 100) × 2 ± 0.15	Square plate, $\Box 100 \times (0.8 \text{ to } 3.0)$		With electrode hole as Fig. 14 (a) (b), $40 \times 20 \times 10$ or full thickness	With electrode hole as Fig. 14 (a) (b), $40 \times 20 \times 10$ or full thickness	With electrode hole as Fig. 14 (c), 40 × ful dia. 26 or under	With electrode hole as Fig. 14 (c), 40 × full dia. 15 or under
) g	Lami- nated tube				7	7		2								
Times of testing	Lami- nated rod		2		<u>-</u>		-								7	2
imes o	Lami- nated sheet			7			2		က	က	က	3		2		
T	Mould- ing mate- rial	7			·				က	က	ო	3	2	8		
	Appli- cable clause	5.8	5.9	5.10.1	5.10.2		5.11.1	5.11.2	5.11.3		5.11.3		5.12		5.12	
	Description	In oil, at specified voltage, for 1 min			For less than 200 mm inside diameter	For not less than 200 mm inside diameter	In 90°C oil	In oil	Flatwise, short-time method, in oil	After immersion in water	Flatwise, 1 min step-by- step method, in oil	After immersion in water	After boiling		After boiling	
	Test item	Withstand voltage		Flatwise withstand	voltage		Edgewise withstand	voltage	Dielectric breakdown	strength			Insulation resistance			

Table 1. (continued)

		Ti	mes o	Times of testing	20			Measured	Expres-
Description	Appli- cable clause	Mould- ing mate- rial	Mould- Lami- ing nated mate- sheet	Lami- nated rod	Lami- nated tube	Size of test piece mm	Preconditioning of test piece	value, unit or method of expres- sion	sion of test results
Volume, surface, 1 min charging	5.13	2				Circular disc, $\phi 100 \times (2 \pm 0.15)$	$C-90^{+4}_{-2} \text{ h/20} \pm 2^{\circ}\text{C/(65}$ $\pm 5) \% \text{ RH}$	MOcm	Mean
			2			Square plate, ☐ 100 × full thickness of 10 or under	$C-90^{+4}_{-2}h/20 \pm 2^{\circ}C/(65$ $\pm 5)$ % RH	MD	value
Mutual induction bridge method	5.14.1	2				Circular disc, \$\phi\$ 100 \times (3 \pm 0.15)	$C-90^{+4}_{-2}h/20 \pm 2 \cdot C/(65$ $\pm 5) \% RH$		
After immersion in water		2				Circular disc, $\phi 100 \times (3 \pm 0.15)$	$D-24 \pm 1 \text{ h/23} \pm 1$ °C		
	5.14.2		2		, _ ,	Square plate, \Box 100 × 3 (full thickness)	$C_{-90}^{+4} h/20 \pm 2^{\circ}C/(65 \pm 5) \% RH$		
After immersion in water			2			Square plate, \Box 100 × 3 (full thickness)	$D-24 \pm 1 \text{ h/23} \pm 1^{\circ}\text{C}$		Mean
Q-meter method	5.14.3	2		•		Circular disc, ϕ 50 × (3 ± 0.15)	$C-90^{+4}_{-2} h/20 \pm 2^{\circ}C/(65$ $\pm 5) \% RH$		value
After immersion in water		23	,		,	Circular disc, $\phi 50 \times (3 \pm 0.15)$	$D-24 \pm 1 \text{ h/23} \pm 1^{\circ}\text{C}$		
			2		.	Square plate, \Box 50 × 3 (full thickness)	$C_{-90} \stackrel{+4}{_{-2}} h/20 \pm 2 \cdot C/(65 \pm 5) \% RH$		
After immersion in water			2			Square plate, \Box 50 × 3 (full thickness)	D-24 ± 1 h/23 ± 1°C		
-	5.15	2				Circular disc, \$\phi\$ 100 \times (3 \pm 0.25)	$C-90^{+4}_{-2}h/20 \pm 2^{\circ}C/(65)$ $\pm 5) \% RH$	ω	Mean
Rockwell hardness	5.16.1	2				Circular disc, \$\phi 50 \times (3 \pm 0.15) doubles			
			2			780 × 6 (piled)	A	1	Mean value
Barcol hardness	5.16.2	2	2			1.5 or over in thickness			

Table 1. (continued)

Expres-								Mean value					
Measured	value, unit or method of expres- sion							(kgf/ mm²)					
	Preconditioning of test piece							¥					
	Size of test piece mm	Square rod > $80 \times (10\pm0.5) \times (4\pm0.2)$	Full thickness (1 to 3) \times (25 \pm 0.5) \times 60	(20 times the thickness) Full thickness (> 3 to 10) x	$(10 \pm 0.5) \times 20$ times the thickness	Full thickness (> 10 to 20) \times (20 \pm 0.5) \times 18 times the thickness	Full thickness (> 20 to 25) \times (35 \pm 0.5) \times 18 times the thickness	Full thickness (10 to 20) × (10 ± 0.5) × 120	Full thickness (> 20 to 25) × (20 \pm 0.5) × 120	$<\phi 8 \times 70$	ϕ (8 to 15) × 120	Machined wall thickness (3 to $10) \times 10 \times 20$ times the thickness	Full dia. (inside dia. to 0.75 times the outside dia.) \times (15 \pm 0.7)
ng	Lami- nated tube											8	8
Times of testing	Lami- nated rod						-			2	8		
imes o	Mould- Lami- ing nated mate- sheet		- 7	- 7		23	8	7					
T	Mould- ing mate- rial	2							· · · · · · · · · · · · · · · · · · ·				
.1	Appli- cable clause	5.17.1	5.17.2							5.17.3		5.17.4	
	Description	$L_{\nu}16\ h\pm0.5$	Perpendicular $L_v16 \ h \pm 0.5$ to laminae					Parallel to $L_v100 \pm 0.5$ laminae		Less than 8 $L_v50 \pm 0.5$ diameter	8 to 15 $L_v100 \pm 0.5$ diameter	Thick wall tube, over 100 inside diameter	Not more than 100 inside diameter
	Test item	Flexural strength and bending	modulus of elasticity		-								

Table 1. (continued)

Expres-	sion of test results		·	Mean value	· · · · · · · · · · · · · · · · · · ·				Mean value	
Measured	value, unit or method of expres- sion			MPa (kgf/ mm ²)					MPa (kgf/	
	Preconditioning of test piece			∢					¥	
	Size of test piece mm	Full thickness (1 to 3) × 25 ± 0.5 × 60 (20 times the thickness)	Full thickness (> 3 to 10) × (10 \pm 0.5) × 20 times the thickness	Full thickness (> 10 to 20) \times (20 \pm 0.5) \times 18 times the thickness	Full thickness (> 20 to 25) × (35 \pm 0.5) × 18 times the thickness	Full thickness (10 to 20) \times (10 ± 0.5) \times 120	Full thickness (> 20 to 25) × (20 \pm 0.5) × 120	Moulded pieces as Fig. 30 $110 \times 45 \times 6.5$	Machined pieces as Fig. 31 Full thickness (0.5 to 10) \times 20 \times 175	For sample over 10 mm in thickness, machine to 10 mm thickness
ng	Lami- nated tube									
Times of testing	Mould- Lami- Lami- ing nated nated mate- sheet rod rial	:								
imes o	Lami- nated sheet	2	2	8	2	23	2			
T	Mould- ing mate- rial							2		
	Appli- cable clause	5.34						5.18.1	5.18.2	
	Description	Vertical to $L_V 16 h \pm 0.5$ laminae				Parallel to $L_v 100 \pm 0.5$ laminae		1		
	Test item	Flexural Ver strength un- lam der heat				Pa ₁		Tensile strength		

Table 1. (continued)

		A 1:	Ë	mes of	Times of testing	gı			Measured	Expres-
Description Cable Ing		Mould- ing mate- rial		Mould- Lami- ing nated mate- sheet	Lami- nated rod	Lami- nated tube	Size of test piece mm	Preconditioning of test piece	value, unit or method of expres- sion	sion of test results
Loading at right angles to 5.19.1 4 direction of moulding pressure		4					Moulded pieces as Fig. 33 (c) ☐ (12.7 ± 0.3) × (25.0 ± 0.3)			
Loading perpendicular to 5.19.2 laminae	5.19.2			2			Machined pieces as fig. 33 (a), piled			
Loading parallel to 5.19.3 laminae	5.19.3			2			Machined pieces as Fig. 33 (b) ☐ (13 ± 0.5) × (25 ± 0.3)			
10 to 20 diameter					2		Machined pieces, full dia. \times (25 \pm 0.3)	¥	MPa (kgf/_	Mean
Over 20 diameter					2		Machined to ϕ 20 concentric circles \times (25 \pm 0.3)		mm.	
Not less than 10 outside dia., not less than 6 inside dia., not less than 2 wall thickness						23	Full dia. (wall thickness 2, machined to concentric circles) × (25 ± 0.3)			
Tubes of thick wall						2	Machined pieces as Fig. 34 \Box (13 \pm 0.3) \times (25 \pm 0.3)			
Loading at right angles to 5.20 2 direction of moulding pressure		23					Fig. 36, with U-notch	V	kJ/m² (kgf·cm/ cm²)	
Parallel to laminae 5.21	5.21			2			Fig. 38, with V-notch in laminae showing side		J/m	Mean value
Perpendicular to laminae				2			Fig. 38, with V-notch in face side	V	{kgf·cm/ cm}	
ϕ 10 steel ball 5.22	5.22		71	2			Fig. 40, thickness (13 ± 0.1) × □ 25	V	N (kgí)	Mean

Table 1. (continued)

ed l	value, unit or method of expres- sion		Occur- rence of	able change		Mean	.c value		1/C Mean		Division	or During re-			Class			Class
	Preconditioning of test piece	V.		•			V V		V	4	A	$E-168 \text{ h/70} \pm 1^{\circ}\text{C}$	¥	$E-168 \text{ h/}70 \pm 1^{\circ}\text{C}$		₩		
	Size of test piece mm	Circular disc, ϕ (50 ± 1) × (3 ± 0.2)	Full thickness × (50 ± 1)	Full dia. × (50 ± 1)	Full dia. × (50 ± 1)	(6.4 ± 0.2) × (12.7 ± 0.2) × 110 min.	$(6.4 \pm 0.2) \times (12.7 \pm 0.2) \times 110 \text{ min.}$	120 × 10 × 10	Full thickness \times 10 \times 10	$120 \times 10 \times 10$	127 × 12.7 × 12.7	Full thickness \times 12.7 \times 12	$0.8 \times (12.7 \ ^{+0.5}_{0}) \times 127$	$0.8 \times (12.7 \ ^{+0.5}_{0}) \times 127$	Full thickness \times (12.7 $^{+0.5}$) \times 127	Full thickness \times (12.7 $^{+0.5}_{0}$) \times 127	0.8 or over \times (12.7 $^{+0.5}$) \times 127	Full thickness $\times (12.7^{+0.5}) \times$
esting umi- Lamirted nated d tube					2													
Times of testing	Lami- nated rod			2											<u> </u>			
imes (Mould- Lami- ing nated mate- sheet		2				7		7	2		2			ಬ	ro.		က
I	Mould- ing mate- rial	7				2		2			2		22	2		<u>-</u>	3	
Appli- cable clause		5.23				5.35		5.25			5.24.1		5.24.5			i	5.24.3	
	Description	Heating for 2 h	Heating for 2 h	Heating for 2 h	Heating for 2 h	ļ			Perpendicular to laminae	Parallel to laminae	Method A		Method B				Method C	
	Test item	Appearance after heating				Deflection temperature under load		Thermal ex-	pansion		Burning re-	Sistance		-				

Table 1. (continued)

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		:	Ţ	mes o	Times of testing	Вu			Measured	Fxnrea-
Test item	Description	Appli- cable clause	Mould- ing mate- rial	Lami- nated sheet	Lami- nated rod	Lami- nated tube	Size of test piece mm	Preconditioning of test piece		sion of test results
Water ab-	l	5.26.1	2				ϕ (50 ± 1) × (3 ± 0.2)			
nond ros		5.26.2		2			Full thickness 🗆 50 ± 1			,
		5.26.3			2		Full dia. × (50 ± 1)	$\int E - 24 \pm 1 \text{ n/30} \pm 2 \text{ C} + \text{des}$	% 	value
		5.26.4				7	Full dia. × (50 ± 1) or Fig. 44			
Boiling water absorption	Boiling for 1 h	5.27	2				ϕ 50 ± 1 × 3 ± 0.2	E-24 ± 1 h/50 ± 2°C + des	%	Mean
Specific gravity	ſ	5.28	-	1	-	1	Suitable shape, dimensions (1 g to 5 g mass)	٧		Mean
							Cut faces to be smooth	•	ļ	value
Resistance to acetone	Applied to phenol resin	5.29	2	2	2	2.	$20 \times 20 \times 20$ or suitable dimensions	A	1	Occurrence of remarkable change in
Resistance to boiling water		5.30	2				ϕ (50 ± 1) × (3 ± 0.2)	A	1	appearance Occurrence of change in ap- pearance and coloration of
Resistance to sulfuric acid	Applied to melamine resin 0.8 ± 0.05 % sulfuric acid	5.31	2				ϕ (50 ± 1) × (3 ± 0.2)	¥	1	water Extent of change in appearance and dis-
Chemical resistance	Changing rate of mass	5.32	2				ϕ (50 ± 1) × (3 ± 0.2)	E-24 ± 1 h/50 ± 2°C	%	Mean value
Remarks: T	The symbols for preconditionir	itioning	are as follows:	s foll	ows:					

immersion in distilled water and O for treatment in oil, and des shows the condition having been allowed to cool A is for as furnished condition, C for constant temperature and humidity, E for constant temperature air, D for in a desiccator containing desiccant.

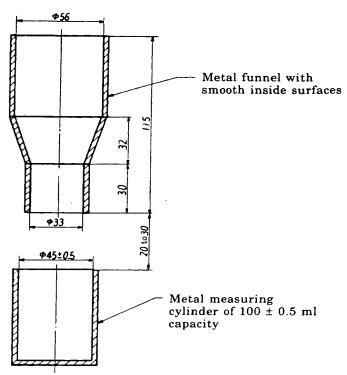
Example: $C-90^{+4}_{-2}$ h/20 \pm 2°C/(65 \pm 5) % RH means to place the test piece in the air of a constant temperature and relative humidity of 20 \pm 2°C and (65 \pm 5) % for 90 $^{+4}_{-2}$ h.

5. Test method

- 5.1 General conditions for tests The general conditions for tests shall be as follows:
- (1) Atmosphere of testing place Tests shall, as a rule, be carried out in a room of $23 \pm 2^{\circ}C$ temperature and (50 ± 5) % relative humidity. When the tests have been carried out under other temperature and humidity than the above, the temperature and humidity shall be noted in addition to the test results. However, for withstand voltage, dielectric breakdown strength, insulation resistance, resistivity, dielectric constant, dielectric dissipation factor and arc-resistance, the tests shall be carried out in a room of $20 \pm 2^{\circ}C$ temperature and (65 ± 5) % relative humidity.
- (2) Maintaining accuracy of apparatus Testing machines and measuring instruments shall be maintained in such a state that tests are reproducible with required accuracies.
- (3) $\frac{\text{Tolerances}}{\text{with the requirements for the items as appropriate.}}$ Unless otherwise specified, the tolerances shall be \pm 5 % of the numerical values.
- (4) Numerical values of test results Determine the numerical values to the place next below the specified, and round them off in accordance with JIS Z 8401.
- 5.2 Apparent density and bulk factor
- 5.2.1 <u>Moulding material</u> (where pouring through funnel is possible) To be carried out as follows:
- (1) Apparatus
- (1.1) Trip balance The balance specified in JIS B 7601 with precision of 0.5 g reciprocal sensibility or better.
- (1.2) Apparent density measuring apparatus Metal ones of the shapes and dimensions as shown in Fig. 1.

Fig. 1. Apparent density measuring apparatus (where pouring through funnel is possible)

Unit: mm



(2) Procedure Measure the mass of the measuring cylinder to the nearest 0.5 g. Close the bottom of the funnel with a flat metal panel, and pour in it 110 ml to 120 ml of well mixed sample. Next, remove the metal panel under the funnel to let the sample freely fall into the measuring cylinder below.

Where the sample is in clods and adhering to the funnel, break them in pieces with a glass stick. Scrape off the part of the sample heaping above the measuring cylinder by moving a straightedge crosswise on the top edge of the cylinder, and measure the mass of the cylinder with the sample in it to the nearest 0.5 g.

(3) <u>Calculation</u> Calculate the apparent density and bulk factor by the following equations:

$$A_1 = \frac{B - C}{D}$$

$$A_2 = \frac{E}{A_1}$$

where, A_1 : apparent density [g/ml (kg/l)]

 A_2 : bulk factor

B: mass of measuring cylinder with sample in it (g)

C: mass of measuring cylinder (g)

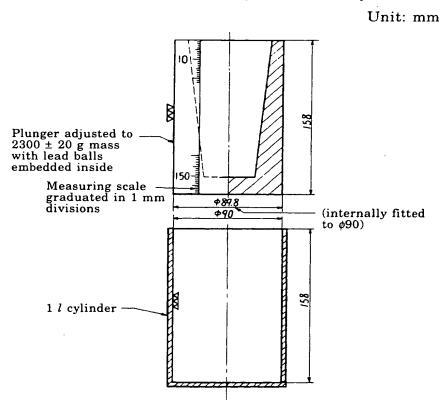
D: capacity of measuring cylinder (ml)

E: density of moulding product [g/ml (kg/l)]

The density of moulding product is the numerical value of specific gravity measured by the method specified in 5.28.

- 5.2.2 <u>Moulding material</u> (where pouring through funnel is impossible) To be carried out as follows:
- (1) Apparatus
- (1.1) Trip balance The balance specified in 5.2.1 (1.1).
- (1.2) Apparent density measuring instrument Metal instrument of the shapes and dimensions as shown in Fig. 2, the plunger being graduated outside in 1 mm divisions from the top to the bottom, and adjusted to 2300 ± 20 g with lead balls.

Fig. 2. Apparent density measuring instrument (where pouring through funnel is impossible)



(2) Procedure Fill 60 ± 0.2 g of the sample in the measuring cylinder making the surface of the sample horizontal. Next, put the plunger into the cylinder and allow it to stand for 1 min, then measure the level of the sample by the scale outside the plunger to the nearest 1 mm.

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(3) <u>Calculation</u> Calculate the apparent density and bulk factor by the following equations:

$$A_1 = \frac{60}{B \times C}$$

$$A_2 = \frac{E}{A_1}$$

where, A_1 : apparent density [g/ml (kg/l)]

 A_2 : bulk factor

B: cross-sectional area of measuring cylinder (cm²)

C: height of sample in measuring cylinder (cm)

E: density of moulding product [g/ml (kg/l)]

In the above, the density of moulding product is the numerical value of the specific gravity as measured by the method specified in 5.28.

5.3 Flow

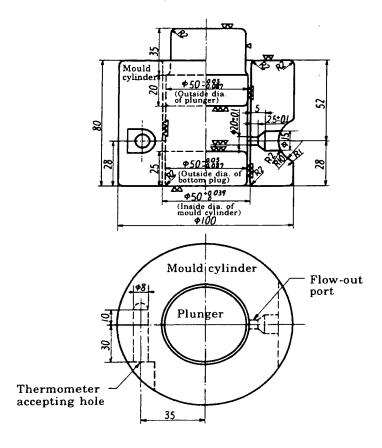
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- 5.3.1 Moulding material (flow by extruding method) To be tested as follows:
- (1) Apparatus
- (1.1) Trip balance The balance specified in 5.2.1 (1.1).
- (1.2) Thermometer A thermometer graduated up to 360°C in 1°C divisions.
- (1.3) Stopwatch Stopwatch graduated in 0.2 s divisions.
- (1.4) Compression moulding machine A moulding machine capable of keeping the metal mould shown in Fig. 3 under the conditions of temperature and pressure given in Table 2.
- (1.5) Metal mould for testing flow by extruding method shapes and dimensions shown in Fig. 3.

Fig. 3. Metal mould for testing flow by extruding method

Unit: mm



Remarks: The material of each part of the mould shall, in accordance with the provisions of JIS G 4051, be as follows:

Plunger : S50C Mould cylinder : S55C

Plunger : S50C Bottom plug : S50C

Procedure Charge the specified mass of the sample in the mould cylinder maintained at the specified temperature, put the plunger into the cylinder within 30 s, apply the specified pressure on the sample and measure the mass of the sample extruded from the flow-out port in the cylinder to the nearest 1 g to represent the flow-out mass (g), and measure the time from the start of pressing to the finish of the flow-out to the nearest 1 s to represent flow-out time (s).

In the above, the charging mass, temperature of metal mould and pressure shall, as a rule, be as given in Table 2.

Where the test was made under other conditions than those in Table 2, they shall be clearly noted in addition to the results.

Table 2. Charging mass, temperature of metal mould and pressure in testing flow by extruding method

Condition	Charging mass	Temperature of metal mould	Pressure (3)
Killd	g	°C	MPa {kgf/cm²}
Phenol resin	40	160 ± 3	A: 9.80 (100) B: 29.0 (300)
Urea resin	35	140 ± 3	20.0 {200}
Melamine resin	35	150 ± 3	20.0 {200}
Diallyl phthalate resin	40	160 ± 3	A: 9.80 {100} B: 20.0 {200}

Notes (3) A: for material of good fluidity

B: for material of little fluidity

5.3.2 Moulding material (flow by circular disc method) To be tested as follows:

- (1) Apparatus
- (1.1) <u>Dimension measuring instrument</u> Graduated in 1 mm divisions.
- (1.2) Thermometer The thermometer specified in 5.3.1 (1.2).
- (1.3) Compression moulding machine A moulding machine capable of maintaining the metal panels shown in Fig. 4 under the conditions of temperature and load given in Table 3.
- (1.4) Metal panels A set of two panels of hard steel, with their faces hard-chrome plated, with shape and dimensions shown in Fig. 4.
- (1.5) Metal cylinder A cylinder approximately 50 mm in inside diameter and approximately 10 mm in height.
- Procedure Put 5 g of the sample on the metal panel maintained at the temperature specified in Table 3, nearly at the centre, using the metal cylinder so as the sample forms a cone, and within 15 s, press-form it under the load and pressing time specified in Table 3. However, where the test was made under other conditions than those in Table 3, they shall be clearly noted.

Measure the major and minor diameters of the glossy part of the formed circular disc by the measuring instrument to the nearest 1 mm, and calculate the average value of the measurements for the elongation (mm) of sample.

Fig. 4. Metal panels for testing flow by circular disc method

Unit: mm

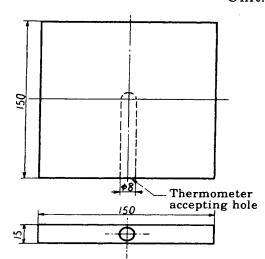


Table 3. Temperature of panels, load and pressing time for testing flow by circular disc method

Condition	Temperature of metal panel	Load	Pressing time
Kind	°C	kN (kgf)	min
Phenol resin	160 ± 3	24.50 {2500}	1
Urea resin	140 ± 3	19.60 (2000)	2
Melamine resin	150 ± 3	19.60 (2000)	2
Diallyl phthalate resin	160 ± 3	24.50 {2500}	1

5.4 Mouldability (moulding material) To be tested as follows:

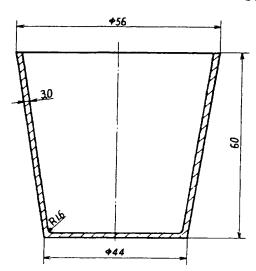
(1) Apparatus

- (1.1) Compression moulding machine A moulding machine capable of maintaining the temperature and the pressure on the metal mould specified in (1.2) as agreed between the parties concerned.
- (1.2) Metal mould A metal mould capable of moulding the sample, as a rule, to the shape and dimensions shown in Fig. 5.
- (2) Procedure Examine the product moulded in the metal mould from the sample to the shape and dimensions shown in Fig. 5, for easiness of separation from the metal mould, the smoothness in appearance, deformation, wrinkles, spots, etc.

The temperature of metal mould, pressure and pressing time shall be as agreed between the parties concerned.

Fig. 5. Dimensions of moulding product in mouldability test





5.5 Dimensions

5.5.1 Laminated sheets To be measured as follows:

(1) Length and width

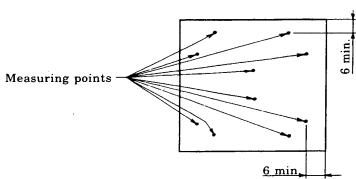
- (1.1) Apparatus Use the steel tape measure of Class 1 (graduated in 1 mm divisions) specified in JIS B 7512 or one with an accuracy at least equivalent.
- (1.2) Procedure Hold the laminated sheet vertical or horizontal and measure, in parallel to the periphery of the sheet, lengthwise and widthwise to the nearest 1 mm.

(2) Thickness

- (2.1) Apparatus Use the micrometer calliper for external measurement specified in JIS B 7502 or at least equivalent provided with a yoke of sufficient size and rigidity to permit accurate measurement of the thickness of the laminated sheet in the centre.
- (2.2) Procedure Hold the laminated sheet vertical or horizontal, measure the thickness at 10 points as shown in Fig. 6 two along each edge, not less than 6 mm inwards, (thus eight with all boundaries) and two in the middle to the nearest 0.01 mm. Make two measurements for one point and take their average as the thickness at the point.

Fig. 6. Measuring points for thickness of laminated sheet

Unit: mm



5.5.2 Laminated rod To be measured as follows:

- (1) Length
- (1.1) Apparatus Use the steel tape measure of Class 1 (graduated in 1 mm divisions) specified in JIS B 7512 or one with an accuracy at least equivalent.
- (1.2) Procedure Hold the laminated rod vertical or horizontal, measure the length in four positions spaced at 90° round the circumference to the nearest 1 mm, and take the average of the measurements as the length.
- (2) Diameter
- (2.1) Apparatus The micrometer calliper for external measurement specified in JIS B 7502, the vernier calliper of the minimum reading value of 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.
- (2.2) Procedure Hold the laminated rod vertical or horizontal, measure the diameters at both ends and in the middle, in four positions spaced at 45°, with the micrometer calliper to the nearest 0.01 mm, and take the average of the measurements as the diameter.

They may be measured with the vernier calliper to the nearest 0.02 mm.

5.5.3 Laminated tube To be measured as follows:

- (1) Length
- (1.1) Apparatus The dimension measuring apparatus used shall be the steel tape measure of Class 1 (graduated in 1 mm divisions) specified in JIS B 7512 or one with an accuracy at least equivalent.
- (1.2) Procedure Hold the laminated tube vertical or horizontal, measure the length in four positions spaced at 90° round the circumference to the nearest 1 mm, and take the average of the measurements as the length.
- (2) Outside diameter
- (2.1) Apparatus
- (2.1.1) Micrometer calliper The micrometer calliper for external measurement specified in JIS B 7502 or one with an accuracy at least equivalent.

- (2.1.2) Vernier calliper The vernier calliper the minimum reading value 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.
- (2.1.3) Steel tape measure The steel tape measure of Class 1 specified in JIS B 7512 or one with an accuracy at least equivalent.
- (2.2) Procedure Hold the laminated tube vertical or horizontal, measure the outside diameters at both ends and in the middle in four directions spaced at 45° by the micrometer calliper to the nearest 0.01 mm and take the average of the measurements as the outside diameter.

They may be measured by the vernier calliper to the nearest 0.02 mm.

With a laminated tube with specially large diameter, the outside diameter may be calculated by the following equation, from the circumferential length measured with the steel tape measure wound around the tube to the nearest 1 mm.

$$D = \frac{L}{\pi} - 2t$$

where, D: outside diameter (mm)

 π : ratio of the circumference of a circle to its

diameter = 3.14

L: circumference of laminated tube (mm)

t: thickness of steel tape measure (mm)

- (3) Inside diameter
- (3.1) Apparatus
- (3.1.1) Dimension measuring instrument The bar-shaped micrometer calliper for internal measurement specified in JIS B 7502 or one with an accuracy at least equivalent, bar-shaped micrometer calliper for internal measurement (extension rod type, 0.01 mm in scale), calliper type micrometer for internal measurement (graduated in 0.01 mm division) or small hole gauge (0.01 mm in scale).
- (3.1.2) Vernier calliper The vernier calliper of the minimum reading value 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.
- (3.2) Procedure For a laminated tube with not less than 50 mm inside diameter, hold it vertical or horizontal, measure the inside diameters in each end plane, in four directions at 45° intervals, with a single unit or extension rod type internal micrometer, to the nearest 0.01 mm, and take the average of the measurements as the inside diameter.

They may be measured with a vernier calliper to the nearest 0.02 mm.

For a laminated tube with not less than 3 mm but less than 50 mm inside diameter, measure the diameters in the same way as that for the laminated tube with not less than 50 mm inside diameter, with a calliper type internal micrometer or small hole gauge.

For a laminated tube with specially large wall thickness, the inside diameter may be calculated by subtracting a length equal to twice the measured wall thickness from the outside diameter.

- (4) Wall thickness and its variation
- (4.1) Apparatus
- (4.1.1) Micrometer calliper A micrometer calliper with convex anvil for pipe thickness measurement (graduated in 0.01 mm divisions).

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- (4.1.2) Dial gauge $\overline{\text{JIS B 7503}}$. The dial gauge of 0.01 mm in scale interval specified in
- (4.1.3) Surface plate The Class 2 surface plate specified in JIS B 7513 or one with an accuracy at least equivalent.
- (4.2) Procedure The wall thickness is the numerical value equal to $\frac{1}{2}$ the difference between the outside and inside diameters measured by (2.2) and (3.2). However, when direct measurement of wall thicknesses of a laminated tube with not less than 6 mm inside diameter is particularly desired, measure the thicknesses with the micrometer calliper with convex anvil for pipe thickness measurement, in each end plane, in eight directions at 45° intervals, to the nearest 0.01 mm, and take the average of the measurements as the wall thickness.

When measuring those of a laminated tube with less than 6 mm inside diameter, place the laminated tube on the surface plate, fit a stick with a diameter slightly smaller than the inside diameter of the tube into it, measure the difference between the outside diameters of the stick and the laminated tube, in eight directions round the circumference at 45° intervals, with the dial gauge to the nearest 0.01 mm, and take the $\frac{1}{2}$ of the average of measurements as the wall thickness.

The variation of the wall thickness of laminated tube is the numerical value of the difference between the maximum and minimum wall thicknesses.

- 5.6 Warping rate or twisting rate (laminated sheets)
- 5.6.1 Apparatus Use those as follows:
- (1) Straightedge The Class B straightedge specified in JIS B 7514 (with rectangular cross section) with an effective length not less than 1.5 m.
- (2) Rule The Class 1 rule (graduated in 1 mm divisions) specified in JIS B 7516 or one with an accuracy at least equivalent.
- 5.6.2 Test piece Use full size laminated sheets.
- 5.6.3 Procedure Hang the laminated sheet vertical by the centre of one edge as shown in Fig. 7 (a) and apply the straightedge parallel to the edge. Apply the straightedge on the concave side of the sheet and measure the maximum clearance between the straightedge and the sheet with the metal rule to the nearest 1 mm.

Repeat similar measurements with other edges and take the maximum clearance as the maximum warping of the laminated sheet.

Hang the laminated sheet vertical by one corner as shown in Fig. 7 (b), apply the straightedge on the horizontal diagonal of the sheet, determine the maximum clearance similarly to the case of maximum warping, and take it as the maximum twisting of the laminated sheet.

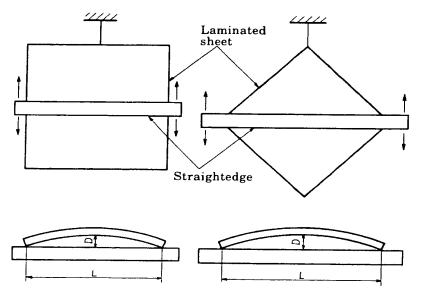
In the case of a laminated sheet not less than 3 mm in thickness, make the measurement with the sheet stood vertical.

Fig. 7. Measuring procedure for warping or twisting of laminated sheets

(a) Measurement of warping

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(b) Measurement of twisting



5.6.4 <u>Calculation</u> Calculate warping or twisting rate W_{1000} (%) by the following equation:

$$W_{1000} = \frac{D_{1000}}{1000} \times 100$$

where, W_{1000} : warping or twisting rate (%)

 D_{1000} : warping or twisting on 1000 mm (mm)

$$D_{1000} = \frac{D}{L^2} \times 1000^2$$

where, D: maximum warping or twisting (mm)

L: span of laminated sheet applied with

straightedge (mm)

5.7 <u>Mould shrinkage coefficient and rate of shrinkage on heating</u> (moulding material)

5.7.1 Apparatus Use those as follows:

- Dimension measuring instrument The micrometer calliper for external (1) measurement specified in JIS B 7502 or a dimension measuring instrument with an accuracy at least equivalent.
- (2) Compression moulding machine A moulding machine capable of keeping the moulds specified in (3) at the temperature and pressure as agreed between the parties concerned.
- A metal mould capable of moulding the material into the (3) Metal mould shape and dimensions shown in Fig. 8.
- A thermometer graduated in 1°C divisions up to 150°C. **(4)** Thermometer
- 5.7.2 Test piece Use a test piece moulded into the shape and dimensions shown in Fig. 8.

Unit: mm **Ф90 480 \$76** Measuring

Fig. 8. Test piece for mould shrinkage coefficient

Remarks: (d_3) and (d_4) show dimensions at the back.

Conduct the tests as follows: 5.7.3 Procedure

Take out the test piece from the moulds (1) Mould shrinkage coefficient immediately after moulding, allow it to stand still for 24 ± 1 h under the conditions (4) of 23 \pm 2°C temperature and (50 \pm 5) % relative humidity, as a rule, and afterwards measure the outside diameters of the rims standing on the face and back of the test piece, in two orthogonal directions for each side, thus taking four measurements in total. Measure the outside diameters of the grooves in the moulds corresponding to the measured points on the test piece to the nearest 0.01 mm, after conditioning the moulds similarly to the test piece.

Moulding conditions (temperature, pressure, time, etc.) are as agreed between the parties concerned.

- Note (4) Where the condition of temperature and humidity differs from the above, it shall be expressly stated.
- (2) Rate of shrinkage on heating Use the test piece specified in 5.7.2, heat it under the condition given in Table 4, then allow it to stand still in the room (4) of 23 ± 2 °C temperature and (50 ± 5) % relative humidity for 3 h to 4 h, and measure the dimensions of the moulding product to the nearest 0.01 mm in the same manner as 5.7.3 (1).

Table 4. Heating conditions for testing for rate of shrinkage on heating

Material	Temperature	Time h		
	°C	Short time	Standard time	
Urea resin moulding material	80 ± 2	48 ± 1	168 ± 2	
Other moulding material	110 ± 3] 10 1 1		

5.7.4 <u>Calculation</u> Calculate the mould shrinkage coefficient and rate of shrinkage on heating by the following equations:

$$MS = \frac{1}{4} \left(\frac{D_1 - d_1}{D_1} + \frac{D_2 - d_2}{D_2} + \frac{D_3 - d_3}{D_3} + \frac{D_4 - d_4}{D_4} \right) \times 100$$

where,

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MS: mould shrinkage coefficient (%)

 d_1, d_2, d_3 and d_4 : outside diameters of rims of test piece

measured on respective measuring lines (mm)

 D_1 , D_2 , D_3 and D_4 : outside diameters of grooves in moulds

corresponding to d_1 , d_2 , d_3 and d_4 , measured

at 23 ± 2°C room temperature (mm)

 PS_{48} or PS_{168}

$$= \frac{1}{4} \left(\frac{d_1 - d_1'}{d_1} + \frac{d_2 - d_2'}{d_2} + \frac{d_3 - d_3'}{d_3} + \frac{d_4 - d_4'}{d_4} \right) \times 100$$

where,

 PS_{48} : rate of shrinkage on heating for 48 h (%)

PS₁₆₈: rate of shrinkage on heating for 168 h (%)

 d_1 , d_2 , d_3 and d_4 : dimensions same as d_1 , d_2 , d_3 and d_4 in

calculating MS (mm)

 d_1 ', d_2 ', d_3 ' and d_4 ': dimensions corresponding to d_1 , d_2 , d_3 and d_4

measured at 23 ± 2°C room temperature after

heating as given in Table 4 (mm)

- 5.8 Withstand voltage (moulding material)
- 5.8.1 Apparatus
- (1) <u>Electrodes</u> A brass spherical electrode well polished with a diameter of 20 mm and a brass disc electrode with a diameter of 25 mm, whose peripheral edge is rounded off to a radius of 2.5 mm.

The surfaces of both electrodes shall be smooth.

- (2) Oil vessel A suitable oil vessel containing the insulating oil specified in JIS C 2320.
- (3) <u>High-voltage breakdown testing apparatus</u> A high-voltage breakdown testing apparatus with not less than 25 kV maximum voltage capable of applying between the electrodes a voltage of a crest factor between 1.34 and 1.48 and of a commercial frequency, 50 Hz or 60 Hz.
 - Remarks: It is preferable to use a testing transformer rated at 2 kVA or over for a testing voltage lower than 50 kV, and one rated at 5 kVA or over for a testing voltage of 50 kV or over. The voltage should be controlled by the use of a variable-ratio autotransformer, resistance-type potential divider, induction voltage regulator or the like, or by the field regulation of the a.c. generator.

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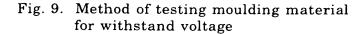
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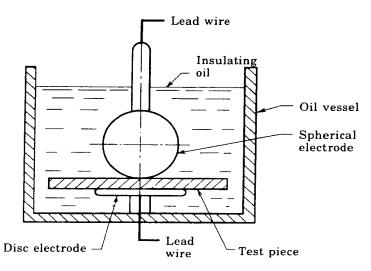
- (4) A.C. voltmeter The a.c. voltmeter of Grade 1.0 specified in JIS C 1102, with the connection of voltmeter in any one way of the following:
 - (a) The a.c. voltmeter is connected to the secondary side of instrument transformer.
 - (b) The electrostatic voltmeter is connected to the secondary side of testing transformer.
 - (c) The a.c. voltmeter is connected to the tertiary winding of testing transformer.
 - (d) The a.c. voltmeter is connected to the primary side of testing transformer. In this case, the ratio of transformation shall not vary due to load variation.
- (5) Thickness measuring instrument measurement specified in JIS B 7502 or one having an accuracy at least equivalent, capable of measuring the thickness of the points about 50 mm inside the periphery of test piece.
- 5.8.2 Test piece Use a test piece moulded to a diameter 60 mm to 100 mm and a thickness of 2 ± 0.15 mm.
- 5.8.3 Preconditioning Precondition the test pieces under C-90 $^{+4}_{-2}$ h/20 \pm 2 °C/ (65 \pm 5) % RH.
- 5.8.4 Procedure Measure the thickness of test piece in the mid part with the thickness measuring instrument to the nearest 0.01 mm. Next, place the test piece finished with conditioning in the oil vessel filled with the insulating oil, hold it between the electrodes by its nearly mid part as shown in Fig. 9 and connect lead wires to the electrodes. Align the both electrodes, when they are holding the test piece, so as their centre lines are in line with each other.

Raise the voltage from zero to the test voltage at a uniform speed as quickly as practicable and examine if the test piece can withstand the test voltage for 1 min. In this case, the test voltage is the voltage obtained by multiplying the specified potential gradient (kV/mm) by the thickness of test piece (mm).

The temperature of insulating oil shall be 20 ± 10 °C.

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- 5.9 Withstand voltage (laminated rods)
- 5.9.1 Apparatus Use those as follows:
- (1) Electrode A well-polished brass spherical electrode with a diameter of 5 mm and a brass disc electrode with a diameter of 10 mm, whose peripheral edge is rounded off to a radius of 1 mm.

The surfaces of both electrodes shall be smooth.

- (2) Oil vessel, high-voltage breakdown testing apparatus and a.c. voltmeter Those specified in 5.8.1 (2), (3) and (4).
- (3) Thickness measuring instrument The micrometer calliper for external measurement specified in JIS B 7502 or one having an accuracy at least equivalent.
- 5.9.2 <u>Test piece</u> Use a test piece cut from the laminated rod at right angels to its axis, with full diameter, both end faces machined at right angels to the axis and the thickness of 2 ± 0.15 mm.
- 5.9.3 Preconditioning Precondition the test piece under C-90 $^{+4}_{-2}$ h/20 \pm 2°C/ (65 \pm 5) % RH.
- 5.9.4 <u>Procedure</u> Measure the thickness of test piece at its centre with the thickness measuring instrument to the nearest 0.01 mm and measure the withstand voltage by the same method as 5.8.4. However, laminated rods having a diameter less than 10 mm and not less than 26 mm are exempted from the test.

- 5.10 Flatwise withstand voltage
- 5.10.1 Laminated sheets To be tested as follows:
- (1) Apparatus
- (1.1) Electrodes, oil vessel, high-voltage breakdown testing apparatus and a.c. voltmeter Those specified in 5.8.1 (1), (2), (3) and (4).
- (1.2) Thickness measuring instrument The micrometer calliper for external measurement specified in JIS B 7502 or one having an accuracy at least equivalent, capable of measuring the thickness of the points about 50 mm inside the periphery of test piece.
- (2) Test piece Use a test piece cut from a part of the laminated sheet into a size about 70 mm to 100 mm in length and width, with full thickness, and shaved off nearly equal thicknesses from both glossy surfaces to leave the middle layer of 2 ± 0.15 mm in thickness.
- (3) Preconditioning $\pm 5)$ % RH. Precondition the test piece under C-90 $^{+4}_{-2}$ h/20 \pm 2°C/(65

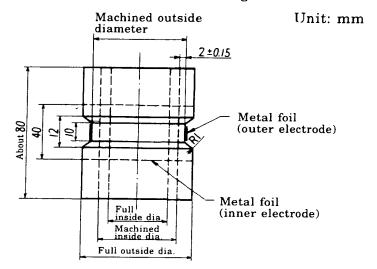
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- (4) Procedure Measure the thickness of test piece at its centre with the thickness measuring instrument to the nearest 0.01 mm and measure the flatwise withstand voltage by the same method as 5.8.4. However, laminated sheets having a thickness less than 2 mm and not less than 10 mm are exempted from the test.
- 5.10.2 Laminated tubes To be tested as follows:
- (1) Apparatus
- (1.1) <u>Electrodes</u> Metal foil electrodes, for laminated tubes less than 200 mm in inside diameter.

The electrodes specified in 5.8.1 (1), for laminated tubes not less than 200 mm in inside diameter.

- (1.2) Oil vessel, high-voltage breakdown testing apparatus and a.c. voltmeter Those specified in 5.8.1 (2), (3) and (4).
- (1.3) Dimension measuring instrument The micrometer calliper for external measurement, the micrometer calliper for internal measurement (barshaped type) specified in JIS B 7502 or ones having an accuracy at least equivalent, such as a bar-shaped internal micrometer calliper (extension rod type), a calliper type internal micrometer calliper (graduated in 0.01 mm divisions) or a small hole gauge (graduated in 0.01 mm divisions).
- (1.4) Thickness measuring instrument A micrometer calliper either face of the points being spherical (graduated in 0.01 mm divisions) capable of measuring the thickness of test piece at the points about 50 mm inside the periphery.
- (2) Test piece
- For laminated tubes less than 200 mm in inside diameter piece cut at right angels to the axis into a length of about 80 mm, with the full diameter, with its wall machined nearly in the middle of the length in parallel with the axis from inside and outside to a thickness of 2 ± 0.15 mm as shown in Fig. 10. In the above the laminated tubes of less than 25 mm in inside diameter or less than 4 mm in wall thickness are machined from outside only, and are not machined from inside.

Fig. 10 Test piece of laminated tube of less than 200 mm in inside diameter for flatwise withstand voltage

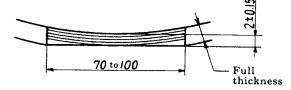


(2.2) For laminated tubes not less than 200 mm in inside diameter A test piece cut into a length and width both about 70 mm to 100 mm, with the full thickness, machined parallel to the axis from outside to a thickness of 2 ± 0.15 mm at the central part as shown in Fig. 11.

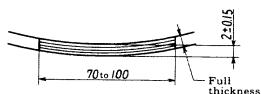
Fig. 11. Test piece of laminated tube of not less than 200 mm in inside diameter for flatwise withstand voltage

Unit: mm

(a) In the case of thick wall







(3) Preconditioning The test piece shall be preconditioned under C-90 $^{+4}_{-2}$ h/ 20 ± 2 °C/(65 \pm 5) % RH.

(4) Procedure

(4.1) Measure the thickness of test piece with the thickness measuring instrument to the nearest 0.01 mm. In the case of laminated tubes of less than 200 mm inside diameter, stick metal foils on the inside and outside surfaces of the test piece with vaseline or the like as electrodes as shown in Fig. 10, connect them to the high-voltage breakdown testing apparatus with lead wires, hang the test piece in the insulating oil of 20 ± 10°C temperature, raise the voltage from zero to the test voltage at a uniform speed as fast as practicable, and examine if the test piece can withstand the voltage for 1 min. In the above, the test voltage is a voltage obtained by multiplying the specified potential gradient (kV/mm) by the thickness of test piece (mm). The metal foil on the inside surface may be substituted by an electroconductive paint.

(4.2) In the case of the test piece of laminated tubes of not less than 200 mm inside diameter, make the inside surface the spherical electrode side on the central part of which a metal foil of projected circular area of 25 mm in diameter is stuck using vaseline or the like, including the case where the width of machined outside surface of the test piece in Fig. 11 (b) becomes less than 25 mm, to make the disc electrode (Fig. 9) side, and measure the flatwise withstand voltage by the same method as 5.8.4. However, laminated tubes having a wall thickness less than 2 mm are not subjected to the test.

5.11 Edgewise withstand voltage

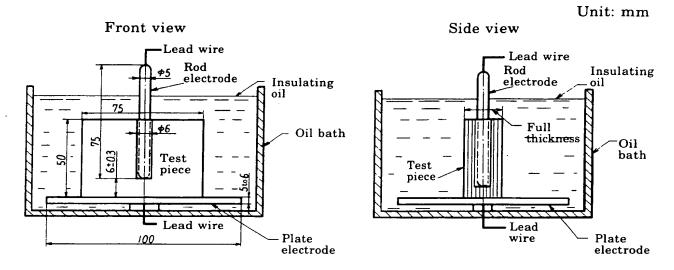
5.11.1 Laminated sheets To be tested as follows:

- (1) Apparatus
- (1.1) Plate electrode A brass plate electrode of about 100 mm in both length and width, and of 5 mm to 6 mm thickness.
- (1.2) Rod electrode A brass rod electrode of 5 mm diameter, 75 mm length having tips of 2.5 mm radius of circle.
- (1.3) Oil vessel An oil vessel capable of holding the insulating oil specified in JIS C 2320 and maintaining it at a constant temperature of $90 \pm 2^{\circ}$ C.
- (1.4) <u>High-voltage breakdown testing apparatus and a.c. voltmeter</u> Those specified in 5.8.1 (3) and (4).
- (1.5) Dimension measuring instrument The micrometer calliper for external measurement specified in JIS B 7502 or one having an accuracy at least equivalent, and a micrometer depth gauge (graduated in 0.01 mm division).
- (2) Test piece Cut out a piece from the laminated sheet with the full thickness, and machine the cut section smooth to a length of 75 mm and a width of 50 mm. Drill a hole of 6 mm diameter in the middle of the longer side section, along the layer as deep as the distance between the electrodes is 6 ± 0.3 mm, and finish the bottom of hole flat with an end mill of 6 mm diameter as shown in Fig. 12. In drilling, be careful not to cause cleaving or else.
- (3) Preconditioning Keep the test piece in an oil bath maintained at 90 ± 2°C for 30 min.
- (4) Procedure Measure the thickness of bottom of hole in the test piece with the micrometer calliper for external measurement and micrometer depth gauge to the nearest 0.01 mm. Next, place the test piece on the plate electrode as shown in Fig. 12, insert the rod electrode into the hole, connect a lead wire to the electrode, allow the test piece to stand in the oil bath maintained at 90 ± 2°C for 30 min to perform the preconditioning of (3), raise the voltage from zero up to the specified test voltage at a uniform speed as fast as practicable while the said temperature is kept, and examine whether the test piece can withstand the voltage for 1 min. However, if any abnormal breakdown is observed, a retest shall be made.

Laminated sheets less than 10 mm or not less than 30 mm in thickness are exempted from the test.

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Fig. 12. Method of testing laminated sheet for edgewise withstand voltage

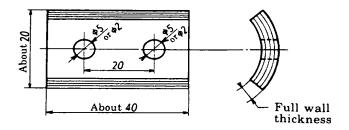


- 5.11.2 Laminated tubes To be tested as follows:
- (1) Apparatus
- (1.1) Electrode Two brass taper pins of 5 mm or 2 mm diameter of class B specified in JIS B 1352, well-polished and having no scars on the surface.
- (1.2) Oil vessel, high-voltage breakdown testing apparatus and a.c. voltmeter Those specified in 5.8.1 (2), (3) and (4).
- (1.3) The taper pin reamer specified in JIS B 4410.
- (2) Test piece Cut out a piece of the shape and dimensions shown in Fig. 13 from the laminated tube, drill two holes perpendicular to laminae, machine them with the taper pin reamer and take the piece for the test piece. In the above the diameter of hole shall be 5 mm for a laminated tube of not less than 10 mm inside diameter and 2 mm for that of less than 10 mm inside diameter.

In the case where it is impossible to cut out a test piece of about 20 mm width from the laminated tube, the width may be of a dimension as sufficient to avoid the discharge between edges.

Fig. 13. Test piece of laminated tube for edgewise withstand voltage

Unit: mm



- (3) Preconditioning The test pieces shall be preconditioned under C-90 $^{+4}_{-2}$ h/ $20 \pm 2^{\circ}$ C/(65 \pm 5) % RH.
- (4) Procedure Press the taper pin into each of the two holes in the conditioned test piece to be the electrodes, connect these to the high-voltage breakdown testing apparatus with lead wires, raise the voltage from zero up to the test voltage at a uniform speed as fast as practicable in the oil bath and examine whether the test piece can withstand the voltage for 1 min.

In the above, the temperature of the insulating oil used shall be 20 \pm 10°C.

5.11.3 Dielectric breakdown strength To be tested as follows:

- (1) Apparatus
- (1.1) High-voltage breakdown testing apparatus
- (1.1.1) Testing transformer To conform to 5.8.1 (4).
- (1.1.2) Automatic circuit-breaker A circuit-breaker designed to interrupt the overcurrent at the fracture of test piece instantly (within 5 cycles). It shall be provided in the primary circuit of the testing transformer.
- (1.1.3) Voltage-controlling device The voltage shall be adjustable by means of a variable-ratio autotransformer, resistance voltage divider, induction voltage regulator or by regulating the field of a.c. generator. An electrical voltage control device is preferable, since it is difficult to keep the speed of voltage-rise within the allowable limits by manual operation.
- (1.1.4) Voltmeter To comply with 5.8.1(4).
- (1.2) Electrodes To comply with 5.8.1 (1).
- (1.3) Oil vessel A suitable oil vessel holding the insulating oil specified in JIS C 2320 (not more than 50 ppm moisture content).

It is preferable that the oil vessel provides an oil-circulating system for maintaining the temperature around the test piece uniform.

The measurement shall be made at $20 \pm 10^{\circ}$ C. However, when the measurement is made at $90 \pm 2^{\circ}$ C as otherwise specified, it shall be noted in addition to the results of measurement.

- (1.4) Thickness measuring instrument To comply with 5.8.1 (5).
- (2) Test piece Use test piece of 80 mm to 100 mm diameter and 2 ± 0.15 mm thickness, or of about 100 mm length and breadth, and 0.8 mm to 3.0 mm thickness. When the thickness is not 2 mm, it shall be noted in addition to the results of measurement.
- (3) Preconditioning The test pieces shall be preconditioned under $C-90^{+4}_{2}$ h/ $20\pm2^{\circ}C/(65\pm5)$ % RH. Dipping shall be done under D-48 ± 2 h/50 ± 3°C, and the test piece shall immediately be wiped perfectly with dry clean acetate cloth, gauze, etc.

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(4) <u>Procedure</u> The testing shall be conducted by the short-time method or step-by-step method.

(4.1) Preparation for voltage application Measure the thickness of test piece at the central five positions to the nearest 0.01 mm. Next, precondition the test piece, then place it in the oil vessel filled with the insulating oil so that its central part lies between the upper and lower electrodes, and fix it with the centre lines of both electrodes aligned. Connect lead wires to the electrodes to form the test circuit.

After removing the bubbles in the insulating oil, measure and record the temperature near the centre of test piece before and after the test.

Check the withstand voltage of the oil by a blank test.

- (4.2) Short-time method Raise the applied voltage from zero with such a uniform speed that causes dielectric breakdown of the test piece in 10 s to 20 s in average. And measure the breakdown voltage at the rapture of the test piece. Obtain the dielectric breakdown voltage by short-time method in MV/m to the first decimal place by dividing the breakdown voltage kV by the mean measured thickness of test piece.
- (4.3) Step-by-step method Select, from Table 5, a voltage nearest to 40 % of the breakdown voltage obtained by the short-time method, and apply this voltage on the test piece for 20 s. If the test piece does not break down, apply the next higher voltage in Table 5 for 20 s, and repeat such procedure until the test piece breaks down.

The raising of the voltage from a step to the next higher one shall be done as rapidly as practicable. This transient time shall be included in the 20 s for the next step.

The voltage of the highest step under which the test piece has not broken down in 20 s is regarded as the dielectric breakdown voltage. Obtain the dielectric breakdown voltage by step-by-step method in MV/m to the first decimal place, by dividing the voltage so measured by the mean measured thickness of the test pieces.

Carry out this test on three test pieces and the strength is stated by the average, but if any test result is deviating from the average by 15 % or more, additional tests equal to the number of deviated results shall be made, and the average shall be recalculated by adding the retested results.

5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	5				
10	11	12	13	14	15	16	17	18	19					
20	22	24	26	28	30	32	34	36	38	40	42	44	46	48
50	55	60	65	70	75	80	85	90	95	100				
110	120	130	140	150	160	170	180	190	200					

Table 5. Voltage steps (effective value $MV \times 10^{-3}$)

5.12 Insulation resistance

5.12.1 <u>Moulding materials</u> To be tested as follows:

(1) Apparatus

- (1.1) <u>Insulation resistance measuring apparatus</u> An apparatus consisting of electrodes, power source, galvanometer, universal shunt, switches, etc. as exampled in Fig. 15.
- (1.1.1) Electrodes The brass taper pins of Class B specified in JIS B 1352, with 5 mm diameter and free from scars on the surfaces.
- (1.1.2) Power source A dry or storage battery at 500 V d.c. voltage. A power source from rectified a.c. may be used provided it is certain that it keeps a certain d.c. voltage.
- (1.1.3) Insulation resistance measuring instrument
- (1.1.3.1) Measuring insulation resistance of not less than 1 $M\Omega$ but less than 10^6 $M\Omega$ (comparison method) The standard resistance shall be of 1 $M\Omega$ manganine or one with an accuracy at least equivalent, and the universal shunt shall be accurate enough for adjusting the deflection and measuring range of the galvanometer.

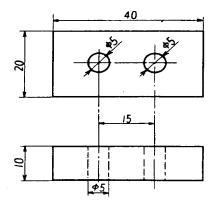
A galvanometer can measure a resistance of less than 10^6 M Ω with \pm 10 % accuracy, if it is highly sensitive with stable zero point as to show 1 mm deflection at 1 m distance by a current of 10^{-10} A.

- (1.1.3.2) Measuring insulation resistance of not more than 5 $M\Omega$ Use the insulation resistance tester specified in JIS C 1302.
- (1.1.3.3) Measuring insulation resistance of not less than 1 M Ω but less than 10⁸ $\frac{M\Omega}{accuracy}$. Use an apparatus having a d.c. amplifier calibrated to \pm 10 %
- (1.1.4) Switches Switches properly insulated and protected.
- (1.2) Taper-pin reamer The taper-pin reamer specified in JIS B 4410.
- (1.3) A suitable apparatus for boiling test pieces.
- (2) Test piece A test piece formed to the shape and dimensions shown in Fig. 14 (a).

Fig. 14. Insulation resistance test piece

Unit: mm

(a) Moulding material



(b) Laminated sheet

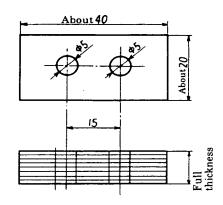
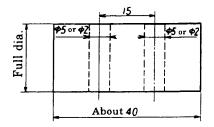
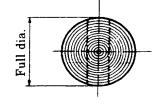


Fig. 14. (continued)

(c) Laminated rod





- (3) Preconditioning ± 5 % RH. Precondition the test piece under C-90 $^{+4}_{-2}$ h/20 \pm 2°C/(65
- Procedure Press the taper-pins into the two holes in the test piece to be the electrodes, and measure the insulation resistance by applying d.c. voltage 500 V using the power source and the insulation resistance measuring instrument.

In measuring the insulation resistance (normal condition), make the measurement after charging the conditioned test piece under the condition specified in 5.1 (1) [20 \pm 2°C temperature, (65 \pm 5) % relative humidity] for 1 min.

In measuring the insulation resistance (after boiling), make the measurement after boiling the conditioned test piece in boiling distilled water for 2 h, cooling it in running fresh water maintained at 20 \pm 10°C for 30 min, taking it out, wiping away the moisture on the surfaces with dry clean gauze, etc., allowing it to stand for 2 min and charging it under the condition specified in 5.1 (1) [20 \pm 2°C temperature, (65 \pm 5) % relative humidity] for 1 min. The insulation resistance measuring instrument used shall be the standard resistance, universal shunt and galvanometer in (1.1.3.1), for an insulation resistance of not less than 1 M Ω but less than 10^6 M Ω , and the insulation resistance tester in (1.1.3.2), for an insulation resistance of not less than 1 M Ω but less than 10° M Ω , the insulation resistance measuring instrument provided with the d.c. amplifier in (1.1.3.3) may be used; and especially for not less than 10^6 M Ω resistance, this instrument should be used.

When measuring the insulation resistance by the comparison method, the measurement shall be made by the following method with the circuit shown in Fig. 15.

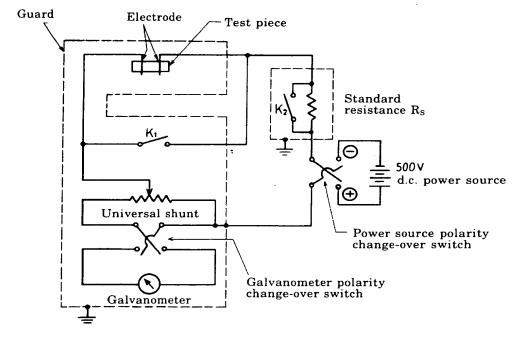
(4.1) Calibration to standard In Fig. 15, close K_1 , open K_2 and throw the power source polarity change-over switch into the polarity desired.

Read the deflection of galvanometer θ_1 when the standard resistance R_S is in series with the galvanometer, with the universal shunt set at a suitable multiplying factor S_1 .

Next, close the galvanometer polarity change-over switch, adjust the multiplying factor of the universal shunt S_2 until the deflection of the galvanometer θ_2 becomes readable, charge the test piece for 1 min and make the measurement.

When measuring a high insulation resistance, provide a guard as shown in Fig. 15 to protect the whole apparatus within the power connections of the electrodes and galvanometer, and insulate them (the whole apparatus within the power connections of the electrodes and galvanometer) perfectly from the guard.

Fig. 15. Insulation resistance measuring apparatus (comparison method)



(4.3) <u>Calculation</u> Calculate insulation resistance by the following equation:

$$R = R_S \frac{S_1 \times \theta_1}{S_2 \times \theta_2}$$

where,

R: insulation resistance $(M\Omega)$

 R_S : standard resistance (M Ω)

 S_1 : multiplying factor of universal shunt when measuring standard resistance R_S

 $heta_i$: deflection of galvanometer when measuring standard resistance R_S (mm)

 S_2 : multiplying factor of universal shunt when connecting test piece

 $heta_2$: deflection of galvanometer when connecting test piece (mm)

5.12.2 Laminated sheets To be tested as follows:

- (1) Apparatus To comply with 5.12.1 (1).
- (2) Test piece Use a test piece cut out from the laminated sheet into the shape and dimensions shown in Fig. 14 (b), with the full thickness, drilled with two holes 5 mm in diameter perpendicular to laminae throughout the thickness.
- (3) Preconditioning $\pm 5)$ % RH. Precondition the test piece under C-90 $_{-2}^{+4}$ h/20 \pm 2°C/(65
- (4) Procedure Make the test in accordance with 5.12.1 (4). Machine the two holes in the test piece with the machine taper-pin reamer and insert the taper pins of 5 mm diameter to be the electrodes.

Paper base laminated sheets of more than 15 mm thickness are exempted from the measurement of insulation resistance (after boiling).

5.12.3 Laminated rods To be tested as follows:

- (1) Apparatus To comply with 5.12.1 (1). Use two taper pins of 2 mm in diameter, when the test pieces are taken from laminated rods less than 10 mm in diameter.
- (2) Test piece Use a test piece cut out from the laminated rod into the shape and dimensions shown in Fig. 14 (c), with the full diameter, drilled with two holes 5 mm in diameter and parallel with each other, perpendicular to the axis. However, when the laminated rod less than 10 mm in diameter is used for a test piece, two holes of 2 mm in diameter shall be drilled.
- (3) Preconditioning Precondition the test pieces under C-90 $^{+4}_{-2}$ h/20 ± 2°C/ (65 ± 5) % RH.
- (4) Procedure Make the test in accordance with 5.12.1 (4). In this case, machine the two holes in the test piece with the machine taper-pin reamer and insert the taper pins to be the electrodes.

Laminated rods of more than 15 mm in diameter are exempted from the measurement of insulation resistance (after boiling), and laminated rods not less than 26 mm in diameter are exempted from the measurement of insulation resistance.

5.13 Resistivity

5.13.1 Moulding material To be tested as follows:

(1) Apparatus

- (1.1) A conductive rubber cut into the shape shown in Fig. 16 by hatching, or a moisture permeable conductive paint.
- (1.2) A power source, insulation resistance measuring instrument and switches in (1.1.2) through (1.1.4) of 5.12.1.
- (1.3) Micrometer calliper The micrometer calliper for external measurement specified in JIS B 7502 or one with an accuracy at least equivalent.

- (1.4) Vernier calliper The vernier calliper of the minimum reading value of 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.
- (2) Test piece Use a test piece moulded into a disc about 100 mm in diameter and 2 mm in thickness.
- (3) Preconditioning Precondition the test pieces under C-90 $^{+4}_{-2}$ h/20 \pm 2°C/ (65 \pm 5) % RH.
- (4) Procedure Measure the thickness of the test piece finished with conditioning, with the micrometer calliper for external measurement accurate to 0.01 mm, and press the conductive rubber upon the test piece in the positions shown in Fig. 16 to be the electrodes.

Alternatively, the electrodes may be provided by painting on the test piece with the moisture permeable conductive paint shown in Fig. 16. In this case, treat the test piece after painting the electrodes paying attention so that the moisture permeable conductive paint does not peal off from the test piece during operation.

Measure the outside diameter of inner circle of face electrode and the inside diameter of ring electrode with the vernier calliper to the nearest 0.02 mm. Make connections shown in Fig. 17 (a) for measuring volume resistivity and shown in Fig. 17 (b) for measuring surface resistivity. Connect this assembly in the position of test piece in the same circuit as that given in 5.12, charge it for 1 min and measure the volume resistivity and surface resistivity.

In the above, carry out the tests under the condition in 5.1 (1) [20 \pm 2°C temperature, (65 \pm 5) % relative humidity].

Fig. 16. Arrangement of electrodes in resistivity test

Unit: mm

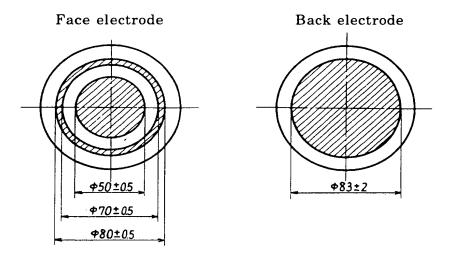
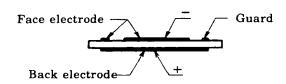
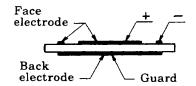


Fig. 17. Connection of electrodes

- (a) Volume resistivity test
- (b) Surface resistivity test





(5) <u>Calculation</u> Calculate volume resistivity and surface resistivity by the following equations:

$$\rho_V = \frac{\pi d^2}{4t} \times R_V$$

$$\rho_S = \frac{\pi \left(D + d\right)}{D - d} \times R_S$$

where, ρ_V : volume resistivity (M Ω cm)

 ρ_S : surface resistivity (M Ω)

d: outside diameter of inner circle of face electrode

(cm)

t: thickness of test piece (cm)

 R_V : volume resistance (M Ω)

D: inside diameter of ring electrode on face (cm)

 R_S : surface resistance $(M\Omega)$

 π : ratio of circle's circumference to its diameter =

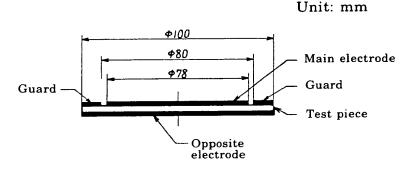
3.14

5.13.2 Laminated sheets To be tested as follows:

- (1) Apparatus To comply with 5.13.1 (1).
- (2) Test piece Use a test piece cut out from the laminated sheet to a length and width of 100 mm each, with the full thickness. In the case of making the test piece from a laminated sheet more than 10 mm in thickness, machine one glossy side to 10 mm thickness.
- (3) Preconditioning Precondition the test piece under C-90 $^{+4}_{-2}$ h/20 ± 2°C/ (65 ± 5) % RH.
- (4) Procedure Make the test in accordance with 5.13.1 (4). In this case, arrange the face electrode on the glossy side of test piece.
- (5) Calculation To comply with 5.13.1 (5).
- 5.14 Dielectric constant and dielectric dissipation factor
- 5.14.1 Moulding material To be tested as follows:
- (1) Apparatus

(1.1) Electrodes As shown in Fig. 18, discs of 78 mm and 100 mm diameters, and a ring of 100 mm outside diameter and 10 mm width, made of metal foil 0.005 mm to 0.05 mm in thickness or moisture permeable conductive paint.

Fig. 18. Arrangement of electrodes in dielectric constant and dielectric dissipation factor tests



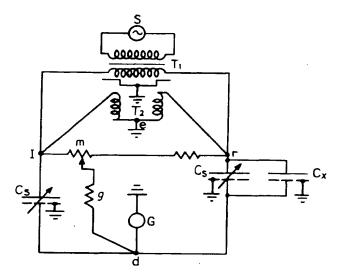
(1.2) Power source of vacuum-tube oscillator or the like (S in Fig. 19) The power source shall be such that its output wave form is close to sine wave as far as practicable (distortion factor is 5 % or less) and capable of supplying the specified voltage stably.

It shall be electrostatically and electromagnetically shielded so as to avoid the direct coupling with the balance detector.

- (1.3) Bridge A bridge having the composition as follows:
- (1.3.1) Shielded transformer (T in Fig. 19) It is used for matching the impedance of the power source with that of the bridge (about 200 Ω), and its winding on the bridge side is shielded with an earthed conductor. In this case, the winding on the bridge side shall be of split balanced type.
- (1.3.2) Ratio arm (T₂ in Fig. 19) Cumulatively connect the primary and secondary windings of a transformer whose turn ratio is 1:1 (with 0.2 % or less error) and whose leakage inductance and winding resistance are as little as practicable, earth the joint as shown by "e" in Fig. 19, and connect the remaining two terminals to "I" and "r" in Fig. 19 respectively to form the ratio arms.
- (1.3.3) Standard variable capacitors (C_s in Fig. 19) They are two air capacitors provided with guards, having a full capacity of about 200 pF, one is used as the standard capacitor and the other as the measuring one, and the sample C_x is connected in parallel with the latter. For the purpose of determining the variation of electrostatic capacity precisely, the capacitors should be such that the capacity variation gradient is gentle in the lower capacity range and steep in the higher capacity range.

Besides, the residual resistance and residual inductance of C_s should be not more than 0.1 Ω and not more than 0.1 μH , respectively.

Fig. 19. Mutual induction bridge method for dielectric constant and dielectric dissipation factor tests (transformer bridge method) measuring circuit



- (1.3.4) Conductance shifter A device as shown in Fig. 19 in which a constant conductance (g) is inserted between "m" and "d", the resistance between "r" and "I" is constant (200 Ω) independent of the position of "m", and the resistance between "I" and "m" is variable from 100 Ω to 0 Ω , that between "r" and "m" from 100 Ω to 200 Ω .
- (1.4) Balance detector A detector that senses only the fundamental wave in the voltage applied to the bridge.
- (1.5) <u>Micrometer calliper</u> The micrometer calliper for external measurement specified in JIS B 7502 or one with an accuracy at least equivalent.
- (1.6) <u>Vernier calliper</u> The vernier calliper of minimum reading value 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.

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- (2) Test piece Use a test piece moulded into a disc 100 mm in diameter and 3 mm in thickness.
- (3) Preconditioning (65 ± 5) % RH. Precondition the test piece under C-90 $^{+4}_{-2}$ h/20 \pm 2°C/
- (4) Procedure Measure the thickness of test piece with the micrometer calliper accurate to 0.01 mm. Attach the electrodes to the test piece finished with conditioning as shown in Fig. 18. When using metal foil as electrodes, coat the test piece with a little quantity of vaseline and quickly attach the electrodes avoiding presence of gap. When using moisture permeable conductive silver paint as electrodes, handle the test piece carefully avoiding the peeling off of the paint from the test piece.

Measure the outside diameter of the main electrode with the vernier calliper to the nearest 0.02 mm and make certain that the ring-form clearance between the main electrode and the guard is 1 mm all round.

Next, connect the conditioned test piece to the position of C_x as shown in Fig. 19, under the test conditions of 5.1 (1) [20 \pm 2°C temperature, (65 \pm 5) % relative humidity], and balance the bridge by adjusting the measuring capacitor C_s and conductance shifter. In this state, measure the value of C_s , conductance between "m" and "d", resistances between "I" and "m" and between "r" and "m" of the conductance shifter in Fig. 19.

(5) <u>Calculation</u> Calculate dielectric constant and dielectric dissipation factor by the following equations:

$$\varepsilon = \frac{C_x}{C_0}$$

$$\tan \delta = \frac{G_x}{2\pi f C_x}$$

where,

ε: dielectric constant

tan δ : dielectric dissipation factor

 C_x : capacity of measuring capacitor C_S when bridge balances (pF)

 C_0 : electrostatic capacity for $\varepsilon = 1$ calculated by the following equation using the area of main electrode and thickness of test piece (pF)

$$C_0 = \frac{r^2}{3.6t}$$

where

r: radius of main electrode

(cm)

t: thickness of test piece (cm)

 G_x : conductance of test piece calculated by the following equation (Ω^{-1}) {S}

$$G_x = g \times \frac{S_V}{100}$$

where,

g: conductance between "m"

and "d" of conductance shifter in Fig. 19 (Ω^{-1}) {S}

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 $\frac{S_V}{100}$: resistance ratio of conductance shifter at balance

point

f: frequency for measurement (Hz), 1 MHz in this test

 π : ratio of circle's circumference to its diameter = 3.14

- Remarks 1. This test method is the mutual induction bridge method (transformer bridge method) applying 1 MHz frequency.
 - 2. The measurement of dielectric constant and dielectric dissipation factor may be made by any one method of the measuring circuits specified in Table 6.

Every measuring circuit given in Table 6 should have an accuracy of \pm 5% for dielectric constant and an accuracy of \pm 5% or measuring accuracy of \pm 1 \times 10⁻⁴ for dielectric dissipation factor.

Table 6. Measuring circuit and range of applied frequency for dielectric constant and dielectric dissipation factor tests

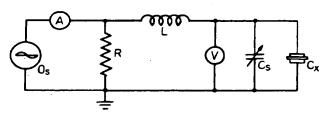
Measuring circuit		Range of applied frequency MHz	
	Resistance ratio capacity bridge	Parallel resistor bridge method	1×10^{-3} to 0.1
Bridge		Series resistor bridge method	2 × 10 ⁻⁴ to 0.1
		High-voltage Schering bridge method	$2.5 imes10^{-5}$ to $1 imes10^{-2}$
method method		Conjugate Schering bridge method	2.5 × 10 ⁻⁵ to 1
	Mutual indu bridge metho	ction bridge method (transformer od)	3.0×10^{-5} to 5
Parallel "T" circuit method		circuit method	0.5 to 50
	Variable conductance method		0.1 to 10
Resonance	Variable sus	ceptance method	0.3 to 30
method	voltage build-up rate method		0.05 to 50
	Variable resistance method		0.1 to 1
Oscillation s	Oscillation starting point method		0.2 to 20

5.14.2 Laminated sheets To be tested as follows:

- (1) Apparatus To comply with 5.14.1 (1).
- (2) Test piece Use a test piece cut out from the laminated sheet to a length and width of 100 mm each, with the full thickness.
- (3) Preconditioning Precondition the test piece under C-90 $^{+4}_{-2}$ h/20 \pm 2°C/ (65 \pm 5) % RH.
- (4) Procedure Make the test in accordance with 5.14.1 (4). In this case, only the laminated sheet of 3 mm thickness shall be tested.
- (5) Calculation To comply with 5.14.1 (5).
- 5.14.3 Voltage build-up rate method (Q-meter method) To conform to 5.14.1 except for the matters prescribed by (1), (2), (3), (4), (5) below.

(1) Apparatus Use the Q-meter having the circuit constitution shown in Fig. 20 and being equipped with the test piece holder shown in Fig. 21.

Fig. 20. Circuit constitution of Q-meter



Os: high-frequency oscillator

A: high-frequency ammeter

R: low resistance

L: tuning coil

 $V\ :\ valve\ voltmeter\ (showing\ deflection\ proportional\ to$

square of input voltage)

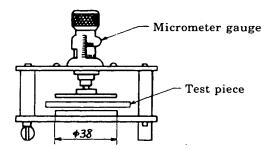
C_S: standard variable capacitor (calibrated beforehand for the capacity including input capacity of valve voltmeter and other stray capacitance by parallel connections)

C_X: test piece holder and test piece

Fig. 21. Test piece holder

Unit: mm

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(2) Test piece For moulding materials, use a test piece moulded into a disc 50 mm in diameter and 3 mm in thickness; for laminated sheets, use one cut off from the sheet to a length and width of 50 mm each, with the full thickness of 3 mm.

Measure the thickness of test piece accurate to 0.01 mm at three points in the area where the electrodes are to be attached, and calculate the average.

(3) Preconditioning Unless otherwise specified, precondition the test piece under $C-90 + \frac{4}{3} h/20 \pm 2^{\circ}C/(65 \pm 5)$ % RH.

For dipping, condition them under $D-24\pm1$ h/23 \pm 1°C and completely wipe off the moisture with dry clean acetate cloth, gauze, etc., and immediately subject them to the test.

(4) Procedure Attach electrodes 38 mm in diameter made of metal foil or conductive paint concentrically on both sides of the preconditioned test piece so as to be on the same axis.

Turn on the power source for the measuring apparatus about 30 min prior to the measurement for stabilization. Set the oscillator O_S at the frequency for measurement f Hz, adjust the output to flow a constant current I through R, watching A, and thus introduce a known low voltage (V = IR) into the resonance circuit.

Set the test piece attached with the electrodes in the test piece holder and connect it in the position C_X . Adjust the standard variable capacitor C_S to cause resonance, watching the valve voltmeter V, and read $oldsymbol{Q}_x$ (or $oldsymbol{V}_x$) and C_{SX} the value of C_{S} at this instance by means of V.

Next, remove the test piece from the test piece holder and adjust and fix the distance between the holder plates to the measured average thickness of the test piece t, by means of the attached micrometer gauge.

Then, increase $\mathbf{C_S}$ until resonance occurs again and read $oldsymbol{Q_0}$ (or $oldsymbol{V_0}$) and the value C_{S0} of C_S .

Calculate dielectric constant and dielectric dissipation factor (5)Calculation by the following equations:

$$\varepsilon = \frac{C_x}{C_0}$$

$$\tan \delta = \frac{G_x}{2\pi f C_x}$$

where,

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 ε : dielectric constant

tan δ : dielectric dissipation factor

 C_x : $C_{S0} - C_{sx} + C_0$ (pF)

 $C_0 : \frac{r^2}{3.6t} (pF)$ $G_x : 2\pi f C_{S0} \left(\frac{Q_0 - Q_x}{Q_0 Q_x} \right) (V)$

 Q_x : $\frac{V_x}{V}$ (voltage build-up rate at resonance, test piece in place)

 $Q_0: rac{V_0}{V}$ (voltage build-up rate at resonance, test piece removed)

r: radius of attached electrode (cm)

 π : ratio of circle's circumference to its diameter = 3.14

t: thickness of test piece (cm)

f: frequency used in measurement (1 MHz)

Remarks: This measuring method has such disadvantages as compared with the bridge method that it gives less measuring accuracy and does not permit the use of guard, but has such advantages that the measuring process is simple and a high frequency can be used, therefore it may be used on agreement between the parties concerned.

5.15 Arc resistance (moulding materials)

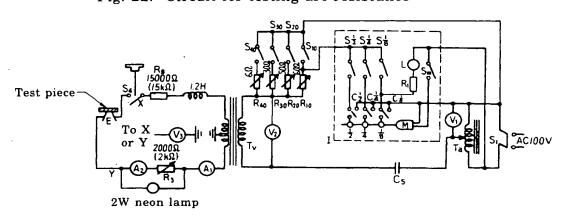
5.15.1 Apparatus

(1) Electric circuit An electric circuit shown in Fig. 22, capable of applying an open-circuit voltage of 12.5 kV {12500 V} with a commercial frequency between the electrodes, in the sequence shown in Table 7.

Table 7. Sequence of applying step voltages in testing for arc resistance

Current	Operation	Duration of on- or off-time	Duration of each step	Total duration
(mA)			(s)	(s)
	$\begin{array}{cccccccccccccccccccccccccccccccccccc$		-	_
10	Turn on $S \frac{1}{8}$ as soon as pilot goes out, start stopwatch	Closed for $\frac{1}{4}$ s, open for $1\frac{3}{4}$ s	60	60
10	Turn on S 1/4	Closed for $\frac{1}{4}$ s, open for $\frac{3}{4}$ s	60	120
10	Turn on $S^{\frac{1}{2}}$	Closed for $\frac{1}{4}$ s, open for $\frac{1}{4}$ s	60	180
10	Turn on S ₁₀	Closed continuously	60	240
20	Turn on S ₂₀	Closed continuously	60	300
30	Turn on S ₃₀	Closed continuously	60	360
40	Turn on S ₄₀	Closed continuously	60	420

Fig. 22. Circuit for testing arc resistance



A: ammeter

V: voltmeter

M: motor

E: electrode section

C: contactor

S: switch

L: pilot lamp

Tv: high-voltage

Ta: variable-ratio

transformer

autotransformer

R: resistor

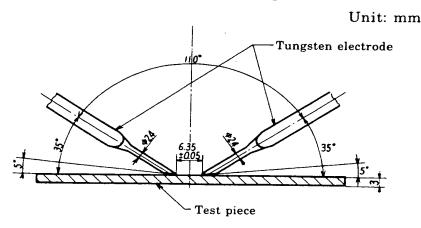
I: switching device

- (2) Electrode Tungsten rods as shown in Fig. 23, each with a length of 20 mm and a diameter of 2.4 mm, and with the tip ground to an inclination of $30 \pm 1^{\circ}$ from its axis.
- (3) Electrode assembly An electrode assembly in which the tips of two electrodes are on the same plane, the axes of the electrodes are both inclined 35° from horizontal, the minor axes of the elliptic faces at the tips are horizontal, their tips are 6.35 ± 0.05 mm apart from each other and the load on the test piece exerted by each electrode can be maintained at 50 ± 5 g.
- (4) Stopwatch A stopwatch graduated in 0.2 s divisions.

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(5) Micrometer calliper The micrometer calliper for external measurement specified in JIS B 7502 or one with an accuracy at least equivalent.

Fig. 23. Electrode assembly for testing arc resistance



- 5.15.2 Test piece Use a test piece moulded into a disc 100 mm in diameter and 3.0 ± 0.25 mm in thickness having smooth test surface.
- 5.15.3 Preconditioning Precondition the test pieces under C-90 $^{+4}_{-2}$ h/20 \pm 2°C/(65 \pm 5) % RH.
- 5.15.4 <u>Procedure</u> Measure the thickness of the test piece in the middle to the nearest 0.01 mm. Make certain whether or not the location of both electrodes on the test piece and the distance between them are correct.

Place the conditioned test piece horizontal, locate the arcing position on the test piece not less than 10 mm apart from the edge and from the parts which were previously subjected to the tests.

Shut out the draught, close the switch S_1 and adjust the variable-ratio autotransformer T_a to give an open-circuit voltage of 12.5 kV between the electrodes. When the switch S_m is closed, the pilot lamp L flickers. Start the stopwatch as soon as the pilot lamp goes out, and simultaneously close the switch $S^{\frac{1}{8}}$. If the test piece is not broken by the arc in 1 min, successively close the switch $S^{\frac{1}{4}}$, and if it is not broken in 1 min, close the switches $S^{\frac{1}{2}}$, S_{10} , S_{20} , S_{30} and S_{40} in 1 min steps, following the sequence of the current steps given in the Table. At the instance when the test piece breaks and the arc goes out, stop the stopwatch and open the switch S_1 . Measure the time from the start of test to the going out of arc to the nearest 1 s, and take it as the arc resistance time (s). In this case, the test is carried out under the conditions of 5.1 (1) [20 \pm 2°C temperature, (65 \pm 5) % relative humidity].

- 5.16 Hardness (moulding material and laminated sheets)
- 5.16.1 Rockwell hardness To be tested as follows:
- (1) Apparatus The Rockwell hardness testing machine specified in JIS B 7726 shall be used.

The testing machine shall be checked prior to testing in accordance with the Standard, for function, load, steel ball indenter, indicator, over-all error and operating time of dashpot and shall be corrected, if necessary.

In the load inspection, the position of top, middle and bottom of service range shall be in the position where the pointer indicates B100 (B=0 on the dial), B30 and around B-50 (pointer indicates B50, after one turn) (a position where the indenter holding spindle is at the lowest point within the range of smooth operation), respectively, when the setting point of the dial is at the right top, and all parts shall operate smoothly.

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The steel ball indenter shall not have a permanent deformation of more than 0.002 mm, be free from rust, and its holder shall be of good workmanship, with its centre line of spindle in line with those of steel ball. The cover shall be fixed in a state to permit free revolution of the steel ball, and the end face of the indenter holding spindle shall be parallel to the anvil face. In general, M-scale is used for hard materials and R-scale for plastic ones. Such a scale on which the hardness number becomes 50 to 115 is preferable.

The operating time of the dashpot shall be adjusted so that with the anvil and indenter set apart form each other without the sample, and making the loading operation of 980 N {100 kgf} test load, the time from the start of operation to the moment the loading lever fully goes down and stops movement is around 5 s.

(2) Test piece It shall be of plate form with flat face and back parallel to each other, having a width of not less than 12 mm and a thickness of not less than 6 mm, as a general rule. Since the thickness may influence the measured value, it shall be noted to the results in the case it is thin. A measuring point shall be not less than 6 mm distant from the edge of the test piece and the adjacent or rear dent.

When measurement is made on a pile of several thin test pieces of the same thickness, it shall be confirmed in advance that they are mutually in contact over the whole surfaces. In this case also, the thickness and the number of test pieces in the pile shall be noted.

of 98 N {10 kgf} on the sample without giving a shock by turning the test piece elevating and lowering wheel until the pointer of the gauge comes within ± 5 divisions of the right top of the dial, the auxiliary pointer being left at the zero point (in the event of the handle's being released reversely, another trial shall be made by changing the position of the sample). Immediately turn the dial of gauge to bring the set position B30 (red scale) to the pointer. Within about 10 s of applying the minor load, apply the major load. After maintaining the major load for 15 s, reduce the load, then 15 s later, read the red scale on the dial to the first decimal place. Count the number of times the pointer passes over zero of the red scale when the

major load is applied. Subtract from this the number of times the pointer passes over zero when the load is reduced. If this difference is zero, record the reading of pointer plus 100 as the hardness number. If the difference is 1, record the reading as it is, and if the difference is 2, record the reading minus 100 as the hardness number. Discard the first measurement. Conduct this test in a room of $23 \pm 2^{\circ}\text{C}$ temperature.

Add the symbol for the scale to the hardness number.

Example: H_RM83, H_RR 76

5.16.2 <u>Barcol hardness</u> The Barcol hardness shall be measured with moulding materials and laminated sheets as follows:

(1) Apparatus A Barcol impressor, Model No. 934-1 shall be used. The indenter shall consist of a hardened steel truncated cone having an angle of 26° with a flat tip of 0.157 mm in diameter. It shall fit into a hollow spindle and be held down by a spring loaded plunger.

The indicating dial shall have 100 divisions, each representing a depth of 0.0076 mm penetration. The higher the reading, the harder the material.

The impressor shall be calibrated in advance with three points as follows: If the values equal to those marked on the standard discs cannot be obtained, the indenter, loading spring or other part is probably not normal, and therefore the instrument shall not be used.

With the plunger upper guide backed out until it lightly touches the loading spring, place the impressor on a glass surface and press down until the point is forced all the way back into the lower plunger guide. At this time, loosen the lock-nut and turn the lower plunger guide in or out so that the indicator indicates 100, and fasten the lock-nut.

The effect of plunger guides on the indication is shown below:

Change by screwing in	Plunger upper guide nut	Lower plunger guide
Change rate of indication	Very little	Very much
Tendency of indication	Falls	Rises
Tendency in relation to scale	The softer, the greater change	Change almost uniformly in relation to hardness number

Next, read the "hard" aluminium alloy disc and the "soft" disc as attached, and readjust by the plunger upper guide nut and lower plunger guide so as the values marked on the standard discs are obtained, and confirm that all the three values equal the marked value.

(2) Test piece The test piece shall be at least 1.5 mm in thickness and have a length and width to enclose an area enough for taking the necessary number of measurements (five) with a boundary allowance of not less than 3 mm inside the periphery.

(3) Procedure Place the test piece on a hard, firm and flat base. When the area of test piece is small, lay a solid plate of the same thickness so that the legs stand on the same horizontal level as the test piece.

Grasp the instrument firmly, with the palm fitted on its back, closely set the indenter and legs perpendicular to the face being tested, press it down by a force of about 40 N to 70 N {4 kgf to 7 kgf}, read and record the maximum indication.

Take care during measurement to avoid sliding of the indenter or occurrence of bending and spring action in the test piece.

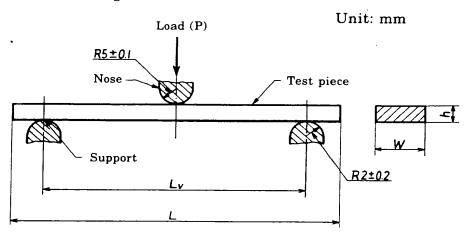
Successively make required number of measurements spacing the measuring points not less than 3 mm apart from each other, and take their average as the hardness of Barcol 934-1. Conduct this test in a room of 23 \pm 2°C temperature.

Add the scale mark "934-1" to the hardness value.

Example: Hardness 68, Barcol 934-1

- 5.17 Flexural strength and bending modulus of elasticity
- 5.17.1 Moulding material To be tested as follows:
- (1) Apparatus
- (1.1) Testing machine A suitable material testing machine that can be operated at a constant rate of crosshead motion. In this case, the allowable error in the load measuring system shall not exceed ± 1% of the standard load of the material testing machine and the load at break shall be equivalent to not less than 15% but not more than 85% of its capacity.
- (1.2) Loading nose A metal nose having an end radius of 5 ± 0.1 mm as shown in Fig. 24.
- (1.3) Supports Metal supports having an end radius of 2 ± 0.2 mm and an adequately adjustable span.
- (1.4) Micrometer calliper The micrometer calliper for external measurement specified in JIS B 7502 or one having an accuracy at least equivalent.
- (1.5) Dial gauge The dial gauge of scale interval 0.01 mm specified in JIS B 7503 or one having an accuracy at least equivalent.
- (1.6) Vernier calliper The vernier calliper giving the minimum reading value 0.02 mm specified in JIS B 7507 or one having an accuracy at least equivalent.

Fig. 24. Flexural test method



(2) Test piece A test piece moulded into a size not less than 80 mm length by 4 ± 0.2 mm height by 10 ± 0.5 mm width as shown in Fig. 25 shall be used.

Fig. 25. Flexural test piece for moulding material

Unit: mm

Load (P)

80 min. (L)

10-05(W)

(3) Procedure Measure the height and width of the test piece accurate to 0.01 mm with the external micrometer calliper. Next, support the test piece with $16h \pm 0.5$ mm span, apply a load at its centre by the loading nose shown in Fig. 24, and measure the load at the break of the test piece to the nearest $1 \text{ N} \{0.1 \text{ kgf}\}$.

If the break has occurred outside the central portion of the test piece divided into three equal lengths, the measurement shall be discarded and a retest shall be made.

Calculate loading speed V (mm/min) by the following equation:

$$V = \frac{h}{2t} \pm 0.2$$

where, V:

V: loading speed (mm/min)

h: height of test piece (mm)

t: time (1 min)

When measuring bending modulus of elasticity, take simultaneous readings of load and deflection frequently to facilitate drawing a load – deflection curve.

(4) Calculation

Calculate flexural strength by the following equation: (4.1)

$$\sigma_{fB} = \frac{3PL_V}{2Wh^2}$$

 σ_{fB} : flexural strength (MPa) {kgf/mm²} where,

P: load at break of test piece (N) {kgf}

 L_V : support span (mm)

W: width of test piece (mm) h: height of test piece (mm)

(4.2)Bending modulus of elasticity shall be calculated by the following equation by drawing a load - deflection curve:

$$E_f = \frac{L_V^3}{4Wh^3} \cdot \frac{F}{Y}$$

where, E_f : bending modulus of elasticity (MPa) {kgf/mm²}

 L_V : support span (mm)

W: width of test piece (mm)

h: height of test piece (mm)

 $\frac{F}{Y}$: gradient of load - deflection curve in linear portion (N/mm) {kgf/mm}

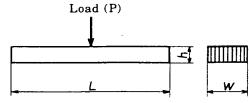
5.17.2Laminated sheets To be tested as follows:

(1)Apparatus To conform to 5.17.1(1).

(2) Use a test piece cut out from the laminated sheet into the shape and dimensions shown in Fig. 26.

Fig. 26. Bending test piece for laminated sheet

(a) Test piece for bending test perpendicular to laminae

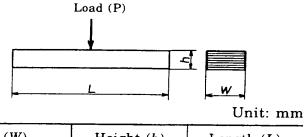


Unit: mm

		Onit. min
Height (h)	Width (W)	Length (L)
1 to 3 incl.	25 ± 0.5	20 <i>h</i> min.
Over 3 to 10 incl.	10 ± 0.5	200 11111.
Over 10 to 20 incl.	20 ± 0.5	18 <i>h</i> min
Over 20 to 25 incl.	35 ± 0.5	10n mm.

In this case, the height (h) shall be the full thickness of laminated sheet.

(b) Test piece for bending test parallel to laminae



Width (W)	Height (h)	Length (L)		
10 to 20 incl.	10 ± 0.5	120 min		
Over 20 to 25 incl.	20 ± 0.5	120 min.		

In this case, W shall be the full thickness of the laminated sheet.

When making a test piece from the laminated sheets of paper-base, fabric-base and asbestos-base exceeding 25 mm in thickness, machine either of glossy sides flat to a thickness of 25 mm.

When making a test piece from the laminated sheets of glass-base and glass mat-base grades exceeding 13 mm in thickness, machine either of glossy sides flat to a thickness of 13 mm.

(3) Procedure To be tested in accordance with 5.17.1 (3). In that, when making the test for bending strength (perpendicular to laminae), the support span (L_v) shall be $16h \pm 0.5$ mm, and for that (parallel to laminae), 100 ± 0.5 mm.

Laminated sheets less than 1 mm thick are exempted from the test for bending strength (perpendicular to laminae), and laminated sheets less than 10 mm thick from the test for bending strength (parallel to laminae).

- (4) <u>Calculation</u> To be done in accordance with 5.17.1 (4).
- 5.17.3 Laminated rods To be tested as follows:
- (1) Apparatus To conform to 5.17.1 (1).
- (2) Test piece A test piece cut from the laminated rod with the full diameter, with a length of 70 mm for rods of less than 8 mm diameter or of 120 mm for not less than 8 mm but not more than 15 mm diameter. However, with a laminated rod of over 15 mm diameter, make the test piece by concentrical machining of the surface around the same axis to a diameter of 15 mm.
- (3) Procedure Make the test in accordance with 5.17.1 (3). In that, measure the diameter of test piece with the external micrometer calliper accurate to 0.01 mm. The support span (L_v) shall be 50 ± 0.5 mm for laminated rods of less than 8 mm diameter and 100 ± 0.5 mm for those of not less than 8 mm but not more than 15 mm diameter.

(4) Calculation Calculate bending strength by the following equation:

$$\sigma_{fB} = \frac{8PL_V}{\pi D^3}$$

where, σ_{B} : bending strength (MPa) {kgf/mm²}

P: load at break of test piece (N) {kgf}

L_v: support span (mm)

D: diameter of test piece (mm)

 π : ratio of circle's circumference to its diameter =

3.14

5.17.4 Laminated tubes To be tested as follows:

- (1) <u>Laminated tubes with inside diameter over 100 mm</u> (of large wall thickness rolled under pressure only)
- (1.1) Apparatus To conform to 5.17.1(1).
- (1.2) Test piece Use a test piece cut out from the full thickness into the shape and dimensions shown in Fig. 27.

Fig. 27. Bending test piece for laminated tubes of large wall thickness

About 10 (W) S 20h min. (L)

(1.3) Procedure Make the test in accordance with 5.17.1 (3). In that, the support span and direction of loading shall be the same as those in the test for bending strength (perpendicular to laminae) of laminated sheet [5.17.2 (3)].

Such laminated tubes from which a test piece of the shape and dimensions of Fig. 27 cannot be taken are exempted from the test.

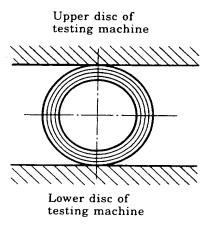
- (1.4) Calculation To be done in accordance with 5.17.1 (4).
- (2) Laminated tubes with inside diameter not more than 100 mm
- (2.1) Apparatus To be as follows:

(2.1.1) Testing machine A material testing machine that can be operated at a constant rate of crosshead motion, having test piece compressing parallel discs shown in Fig. 28. In this case, the allowable error in the load measuring system shall not exceed ± 1 % of the standard load of the material testing machine and the load at break shall be equivalent to not less than 15 % but not more than 85 % of its capacity.

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Fig. 28. Bending test method of laminated tube



- (2.1.2) Micrometer calliper The micrometer calliper for external measurement specified in JIS B 7502 or one having an accuracy at least equivalent.
- (2.1.3) The micrometer calliper for internal measurement (bar-shaped type) specified in JIS B 7502, a small hole gauge (graduated in 0.01 mm divisions), a calliper type internal micrometer (graduated in 0.01 mm divisions) or one with an accuracy at least equivalent.
- (2.1.4) Vernier calliper The vernier calliper giving the minimum reading value 0.02 mm specified in JIS B 7507 or one having an accuracy at least equivalent.
- (2.2) Test piece Use a test piece cut out from the laminated tube with full diameter at right angels to its axis to a length of 15 mm. In this case, if the ratio of inside diameter to outside diameter is less than 0.75, machine the outside concentrically to the axis to make the ratio 0.75.
- Procedure Measure the outside and inside diameters of test piece with the external micrometer calliper and internal micrometer calliper accurate to 0.01 mm, and measure the length with the vernier calliper to the nearest 0.02 mm. Next, place the test piece between the two compressing discs of material testing machine as in Fig. 28, apply a load perpendicular to the laminae and measure the load at the break of test piece to the nearest 10 N {1 kgf}. In the case of laminated tubes formed by rolling, compress at the joints of moulds in the forming. Laminated tubes having a ratio of inside diameter to outside diameter of not less than 0.95 are exempted from the test.
- (2.4) <u>Calculation</u> Calculate bending strength by the following equation:

$$\sigma_{fB} = \frac{3P (D + d)^2}{\pi L d (D - d)^2}$$

where, σ_{fB} : bending strength (MPa) {kgf/mm²}

P: load at break of test piece (N) {kgf}

D: outside diameter of test piece (mm)

d: inside diameter of test piece (mm)

L: length of test piece (mm)

 π : ratio of circle's circumference to its diameter =

Unit: mm

3.14

5.18 Tensile strength

5.18.1 Moulding material To be tested as follows:

(1) Apparatus

- (1.1) Testing machine A material testing machine that can be operated at a constant rate of crosshead motion. In this case, the allowable error in the load measuring system shall not exceed \pm 1% of the standard load of the material testing machine and the load at break shall be equivalent to not less than 15% but not more than 85% of its capacity.
- (1.2) Test piece holders Two test piece holders made of metal, that can surely hold the test piece, having the shape and dimensions shown in Fig. 29. These holders are needed to hold the test piece on the upper movable disc and lower fixed disc of the material testing machine. Attach the holders to the movable disc and fixed disc of the material testing machine, so that the longitudinal axis of test piece and the imaginary centre line of two holders coincide in the same vertical line when applying a load to the test piece. It is necessary that the holders do not permit the slipping of test piece and do not deflect until the test piece breaks.
- (1.3) Micrometer calliper The micrometer calliper for external measurement specified in JIS B 7502 or one having an accuracy at least equivalent.
- (1.4) Vernier calliper The vernier calliper giving the minimum reading value 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.

Fig. 29. Tension test piece holder for moulding material

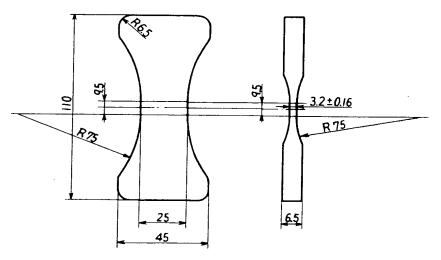
72.5 100 12.5 100 Spacing plate

- (2) Test piece Use a test piece moulded into the shape and dimensions shown in Fig. 30.
- (3) Procedure Measure the width and thickness of parallel part of test piece with the external micrometer calliper accurate to 0.01 mm. Measure other dimensions with the vernier calliper and confirm if they are within the specified limits.

Next, after attaching the test piece to the two holders given in Fig. 29, adjust them so that the points of application of force are in the centre line of test piece. Apply a load on the test piece at a rate of 5 ± 1 mm/min, and measure the load at the break of the test piece nearly at the centre to the nearest 10 N $\{1\ kgf\}$. If the break has taken place outside the mid parallel portion, the measurement shall be discarded and a retest shall be made.

Fig. 30. Tension test piece for moulding material

Unit: mm



(4) <u>Calculation</u> Calculate tensile strength by the following equation:

$$\sigma_{tB} = \frac{P}{A} = \frac{P}{t \times W}$$

where,

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 σ_{tB} : tensile strength (MPa) {kgf/mm²}

P: load at break of test piece (N) {kgf}

A: least original cross-sectional area of test piece

 (mm^2)

t: thickness of test piece (mm)

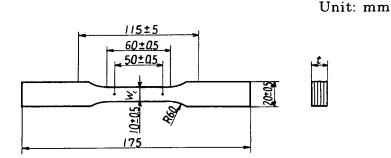
W: width of test piece in mid portion (mm)

5.18.2 Laminated sheets To be tested as follows:

(1) Apparatus To conform to 5.18.1 (1). In this case, the gripping devices shall be two metal parts that can tightly hold the test piece.

(2) Test piece Use a test piece cut from the laminated sheet with the full thickness and machined to the shape and dimensions given in Fig. 31. When the thickness is over 10 mm, machine it to 10 mm. The gauge marks shall be put with crayon or ink, but never by scratching or punching. In machining, care shall be taken not to cause such defects as flaws and scars on the surfaces and end planes of test piece, and if the machined surfaces are rough, finish them smooth with a fine file, emery or the abrasive paper specified in JIS R 6252.

Fig. 31. Shape of tension test piece for laminated sheet



t: thickness of sample plate (mm), in the case of plate over 10 mm, thickness machined to 10 mm.

 W_c : width of test piece in mid portion (mm)

- (3) Procedure Measure the width (W_c) and thickness (t) of test piece at the centre of gauge length with the external micrometer calliper accurate to 0.01 mm. Measure other dimensions with the vernier calliper and confirm if they are within the specified limits. The distance between the gripping devices shall be 115 ± 5 mm. Attach the gripping devices so that the points of application of force are in the centre line of test piece. Apply a load on the test piece at a rate of 5 ± 1 mm/min and measure the load at the break of the test piece in nearly mid portion to the nearest $10 \text{ N } \{1 \text{ kgf}\}$. If the test piece has broken outside the gauge length, the measurement shall be discarded and a retest shall be made. Laminated sheets not more than 1 mm in thickness are exempted from the test.
- (4) Calculation Calculate tensile strength by the following equation:

$$\sigma_{tB} = \frac{P}{A} = \frac{P}{t \times W_c}$$

where, σ_{tB} : tensile strength (MPa) {kgf/mm²}

P: load at break of test piece (N) {kgf}

A: least original cross-sectional area of test piece (mm²)

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t: thickness of test piece (mm)

 W_c : width of test piece in mid portion (mm)

5.19 Compressive strength

5.19.1 Moulding material To be tested as follows:

(1) Apparatus

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- (1.1) Testing machine A suitable material testing machine that can be operated at a constant rate of crosshead motion. In this case, the allowable error in the load measuring system shall not exceed ± 1 % of the standard load of the material testing machine and the load at compressive break shall be equivalent to not less than 15 % but not more than 85 % of its capacity.
- (1.2) Compression testing tool A testing tool constructed as shown in Fig. 32. For the bearing block, use a steel plate with its upper face hardened and polished as flat as possible, and set it in the frame holding the plunger. The lower face of plunger shall be polished as flat as possible and shall be perfectly parallel with the upper face of bearing block and there shall be no gap not less than 0.02 mm when it contacts the bearing block. The top of plunger shall have a round cavity to surely accept the hardened steel ball.

Between the plunger and frame, put a hardened, polished and lapped bush, and apply suitable lubricating oil to ensure smooth motion.

Fit the compression testing tool to the centre of crossheads of material testing machine.

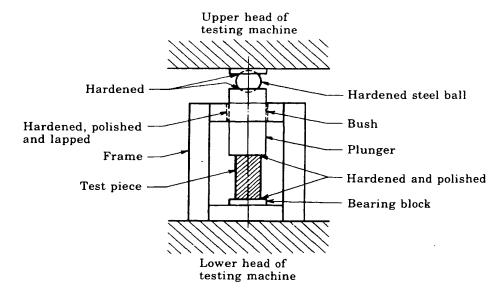


Fig. 32. Compression testing tool

- (1.3) Micrometer calliper The micrometer calliper for external measurement specified in JIS B 7502 or one having an accuracy at least equivalent.
- (1.4) Vernier calliper The vernier calliper giving the minimum reading value 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.

(2) Test piece A test piece moulded to a size of 25.4 ± 0.3 mm length by 12.7 ± 0.3 mm width by 12.7 ± 0.3 mm thickness as shown in Fig. 33 (c), or cut from a piece moulded to a size of approximately 127 mm length by 12.7 ± 0.3 mm width by 12.7 ± 0.3 mm thickness and machined to 25.4 ± 0.3 mm length. In every case, the parallelism of the planes to be applied with load shall be within 0.1 mm.

Fig. 33. Compression test piece

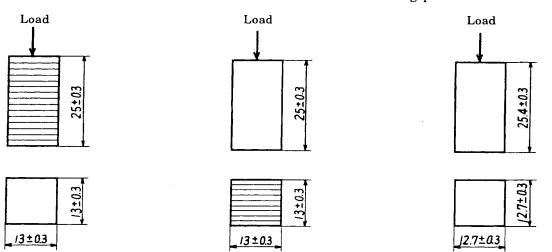
Unit: mm

Test piece for laminated sheets

Test piece for moulding material

(a) Loading perpendicular to laminae

- (b) Loading parallel to laminae
- (c) Loading at right angles to direction of moulding pressure



(3) Procedure Measure the length, width and thickness of test piece with the external micrometer calliper accurate to 0.01 mm. Measure the height with the vernier calliper to 0.02 mm and confirm the parallelism.

Place the test piece on the bearing block of compression testing tool as shown in Fig. 32, and adjust it so that the centre lines of test piece and compression tool are in the same vertical line and the upper and lower faces of test piece are exactly in parallel with the bearing faces of compression testing tool. Apply a load on the plunger of compression testing tool by the material testing machine at a rate of crosshead motion of 1 ± 0.5 mm/min, and measure the load at the break of test piece to the nearest 50 N $\{5 \text{ kgf}\}$.

(4) <u>Calculation</u> Calculate compressive strength by the following equation:

$$\sigma_{cB} = \frac{P}{A}$$

where, σ_{cB} : compressive strength (MPa) {kgf/mm²}

P: load at break of test piece (N) {kgf}

A: original cross-sectional area of test piece

subjected to compression (mm²)

5.19.2 Laminated sheets To be tested as follows:

- (1) Apparatus To conform to 5.19.1 (1).
- (2) Test piece Use a test piece cut from the laminated sheet to a length and width of 13 ± 0.3 mm each, and thickness of 25 ± 0.3 mm as shown in Fig. 33.

In the case of making the test for compressive strength (perpendicular to laminae), if the laminated sheet is not less than 13 mm but less than 25 mm in thickness and the above stated test piece cannot be taken, cut one or more liners having the same shape and dimensions as the test piece from the same laminated sheet, pile them up on the disc and place the test piece on them to the aggregate thickness of 25 ± 0.3 mm. Adjust the total thickness by machining each of the liners to an equal thickness as far as possible.

With laminated sheets of over 25 mm thickness, machine them from either of glossy faces to a thickness of 25 ± 0.3 mm as shown in Fig. 33 (a).

In the case of making the test for compressive strength (parallel to laminae), with laminated sheets of over 13 mm thickness, machine them from either of glossy faces to a width of 13 ± 0.3 mm as shown in Fig. 33 (b).

(3) Procedure Make the test in accordance with 5.19.1 (3). In that, when making the test perpendicular to laminae, apply the load perpendicularly to laminae, and when making it in parallel to laminae, apply the load in parallel to laminae.

Laminated sheets less than 13 mm in thickness are exempted from the test.

- (4) Calculation To be done in accordance with 5.19.1 (4).
- 5.19.3 Laminated rods To be tested as follows:
- (1) Apparatus To conform to 5.19.1 (1).
- (2) Test piece For laminated rods of not less than 10 mm but not more than 20 mm diameter, use a test piece cut out at right angles to the axis to a length of 25 ± 0.3 mm with the full diameter. For laminated rods exceeding 20 mm in diameter, use a test piece machined from the surface concentrically with the axis to a diameter of 20 mm and cut out at right angles to the axis to a length of 25 ± 0.3 mm.
- (3) Procedure Make the test in accordance with 5.19.1 (3). In this case, measure the diameter of test piece with the external micrometer calliper accurate to 0.01 mm and apply the load along the axis of test piece.

Laminated rods of less than 10 mm diameter are exempted from the test.

(4) Calculation To be done in accordance with 5.19.1 (4).

5.19.4 Laminated tubes

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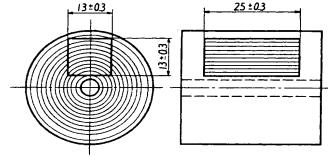
(1) Apparatus Use, in addition to those specified in 5.19.1 (1), the micrometer calliper for internal measurement (bar-shaped type) specified in JIS B 7502, or a small hole gauge (graduated in 0.01 mm divisions), a calliper type internal micrometer (graduated in 0.01 divisions) or internal micrometer (graduated in 0.01 mm divisions, extension rod type).

(2) Test piece For a laminated tube of not less than 10 mm outside diameter, not less than 6 mm inside diameter and not less than 2 mm wall thickness, use a test piece cut out at right angels to the axis to a length of 25 ± 0.3 mm with full diameter, or one cut from the tube having been machined concentrically with its axis from outside to a wall thickness of not less than 2 mm at right angles to the axis to a length of 25 ± 0.3 mm.

With a laminated tube of large wall thickness, it is allowed to cut out a test piece of the shape and dimensions shown in Fig. 34 from the wall thickness.

Fig. 34. Compression test piece for laminated tubes with large wall thickness

Unit: mm



(3) Procedure Make the test in accordance with 5.19.1 (3). In that, measure the outside and inside diameters of the test piece with full diameter or made by concentrical machining, with the external micrometer calliper, bar-shaped internal micrometer or the like accurate to 0.01 mm and apply the load along the axis.

A laminated tube of less than 10 mm outside diameter, less than 6 mm inside diameter or less than 2 mm wall thickness, and the one on which the test is difficult due to its large outside diameter are exempted from the test.

- (4) Calculation To be done in accordance with 5.19.1 (4).
- 5.20 Charpy impact strength (moulding material)
- 5.20.1 Apparatus Use the following:
- (1) Testing machine Use a Charpy impact testing machine as shown in Fig. 35, having the construction as follows:
- (1.1) The pointer shall give a reading accurate to 1 % of the weighing capacity, the centre of angle reading dial, centres of pointer and pendulum shafts shall be perfectly aligned in a line, and the frictional loss of pointer shaft shall have no irregularity irrespective of the position of pointer.

The capacity of testing machine used shall be such that the breaking energy corresponds to not less than 15 % but not more than 85 % of the capacity.

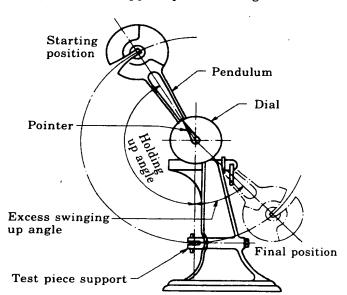


Fig. 35. Charpy impact testing machine

(1.2) The striking edge shall have an included angle of 30 ± 1° and shall be rounded to a radius of 2 ± 0.2 mm, shall uniformly touch the struck face of test piece, its centre shall be within ± 5 mm of the centre of percussion of pendulum, but shall as far as practicable coincide with it as shown in Fig. 36, and the edge shall pass through the centre between the anvils. The linear velocity of striker at the instant of impact shall be 3 m/s.

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(1.3) As shown in Fig. 36, the anvils shall have a corner radius of 1 ± 0.2 mm and a span of 60 ± 0.2 mm, forming no side walls.

In addition, the anvils shall be so aligned that when the pendulum hangs freely, the striking edge just uniformly touches the test piece set in place, as shown in Fig. 36.

- (1.4) For the calculation of impact strength, the testing machine shall be prepared with its specific energy table as follows:
- (1.4.1) Measure the total mass of pendulum system (W_0) , which consists of the striker, bearing, one of the two ball bearings, shaft, pointer and the whole moving part of their fixing metals, to the nearest 1 g.
- (1.4.2) Adjust the machine so that the frictional loss (J) {kgf·cm} when the pendulum provided with the pointer is freely swung from the maximum holding up angle of 150°, becomes about 1 % (about 40 mJ) {0.4 kgf·cm} (corresponding to about 2° angle) or less, though it differs depending on the weighing capacity.
- (1.4.3) Measure the centre of gravity of pendulum attached with its parts specified in (1.4.1), by supporting it on a knife edge. Determine the position where it rests horizontal, further do the same procedure changing the relative position and determine the centre of gravity from the supporting points so obtained.
- (1.4.4) Obtain the distance between the centre of gravity of pendulum so obtained and the centre of shaft (R) to the nearest 0.1 mm.

- (1.4.5) As the weighing capacity $[W_0R (1 \cos \alpha)]$ (J) {kgf·cm}, take the value corresponding to the maximum holding up angle (α) of 150°.
- (1.4.6) For the period of oscillation of pendulum (T), measure the time for 100 cycles of the pendulum by releasing it from a position 5° from the vertical to allow free swing and calculate the quotient of the time in seconds divided by 100 to the third decimal place.
- (1.4.7) Calculate the distance between the centre of shaft of pendulum and the centre of percussion (L_0) by the following equation to the second decimal place.

$$L_0 = g T^2 / 4\pi^2 = 24.82T^2$$

where,

 L_0 : distance between centre of shaft of pendulum and centre of percussion (cm)

g: gravitational acceleration (980 cm/s²)

T: period of pendulum oscillation (s)

 π : ratio of circle's circumference to its diameter

- (1.4.8) The difference between the distance of centre of shaft of pendulum from the striking point (centre of striking edge) (L) and the distance of the same from the centre of percussion defined in (1.4.7) (L_0) , $(L-L_0)$, shall be not more than 5 mm.
- (1.4.9) Calculate the striking speed by the following equation:

$$V = \sqrt{2 g L_0 (1 - \cos \alpha)}$$

where.

V: striking speed (cm/s)

q: gravitational acceleration (980 cm/s²)

 L_0 : distance between centre of shaft of pendulum

and centre of percussion (cm)

 α : pendulum holding up angle

(1.4.10) Calculate the absorbed energy (E) by the following equation:

$$E = 1/2W_0R (3 \cos \beta - \cos \beta' - \cos \alpha - \cos \alpha')$$

where,

E: absorbed energy (J) {kgf·cm}

 W_0 : mass of pendulum system (kg)

R: distance between centre of gravity of pendulum

and centre of shaft (cm)

 α : pendulum holding up angle

lpha': mean excess swinging up angle of pendulum

when released from angle α

eta: excess swinging up angle after breaking test

piece from holding up angle of α

eta': mean excess swinging up angle from holding up

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angle of β

- (1.4.11) To ease the calculation of impact strength, prepare a table showing the relation between the excess swinging up angle after breaking test piece β and the absorbed energy (E).
- (2) <u>Micrometer calliper</u> The micrometer calliper for external measurement specified in JIS B 7502 or one with an accuracy at least equivalent.
- (3) Vernier calliper The vernier calliper giving the minimum reading value 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.
- 5.20.2 <u>Test piece</u> Use a test piece having the shape and dimensions shown in Fig. 36. The notch region (hereafter referred to as "notch") shall, as a rule, be formed by machining. The bottom of notch shall be finished smooth and be free from any scars or the like which might adversely affect the test.

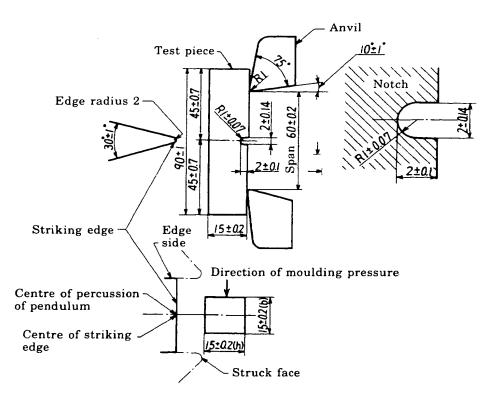
The angle included by the symmetrical sides of the notch and the longitudinal axis of the test piece shall be within $90 \pm 2^{\circ}$.

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In the case of a test piece made by compression moulding, the notch shall be machined on the parallel side to the direction of compression moulding.

Fig. 36. Charpy impact test piece

Unit: mm



5.20.3 Procedure Measure the width and depth of test piece at the notch accurate to 0.01 mm, measure the dimensions of rectangular parallelepiped to the nearest 0.02 mm and confirm if each face is parallel to its opposite.

Confirm that the testing machine is installed horizontal in both fore-and-aft and lateral directions.

Align the centre line of notch in test piece with the centre of span of anvils as shown in Fig. 36, and set the test piece accurately to be struck on the opposite side of notch. Confirm that the struck face just uniformly touches the striking edge of the freely hanging pendulum.

Hold the pendulum and pointer in the starting position by the stopper, next release it quickly to strike the test piece at a linear velocity of 3 m/s at the instant of impact. Read the excess swinging up angle β when the test piece is broken by one strike and determine the energy (E) corresponding to β from the energy table [see 5.20.1 (1.4)].

5.20.4 <u>Calculation</u> Calculate Charpy impact strength by the following equation:

$$A_{KC} = \frac{E}{b \times h}$$

where, A_{KC} : Charpy impact strength (kJ/m²) {kgf·cm/cm²}

 ${\it E}$: energy when test piece is broken by one strike

(kJ) {kgf·cm}

b: width of test piece at notch (cm)

h: minimum thickness of test piece at bottom of

notch (cm)

5.21 Izod impact strength (laminated sheet)

5.21.1 Apparatus Use the following:

- (1) Testing machine Use an Izod impact testing machine as shown in Fig. 37, having the construction as follows:
- (1.1) The pointer shall indicate accurate to 1 % of the weighing capacity, the centre of angle reading dial, centres of pointer and pendulum shafts shall be perfectly aligned in aline and the frictional loss of pointer shaft shall have no irregularity irrespective of the position of pointer.

The capacity of testing machine used shall be such that the breaking energy corresponds to not less than 15 % but not more than 85 % of the capacity.

(1.2) The striking edge shall have 0.8 mm radius, shall uniformly touch the struck face and the centre of percussion of pendulum shall coincide with the tangent line of striking edge to the test piece (in any case the difference shall not be over 5 mm) and pass the point 22 ± 0.5 mm above the top of vise, as shown in Fig. 39.

The linear velocity of striking edge at the instant of impact shall be 3.3 m/s.

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(1.3) The test piece holding vise shall be such that the top faces of fixed and movable jaws are in the same plane and horizontal. Top faces of jaws shall be at right angles to the faces touching the test piece (corner radius shall be not more than 0.8 mm).

A vice, when holding the test piece in place, the struck face and its top face include 90°, and the striking edge of freely hanging pendulum can just uniformly touch the struck face.

For the calculation of impact strength, the testing machine shall be (1.4)prepared with specific energy table [see 5.20.1 (1.4)].

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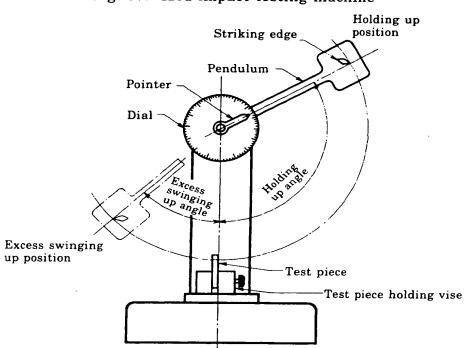


Fig. 37. Izod impact testing machine

- (2)Micrometer calliper The micrometer calliper for external measurement specified in JIS B 7502 or one with an accuracy at least equivalent.
- (3) Vernier calliper The vernier calliper giving the minimum reading value 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.
- 5.21.2 Test piece Fig. 38 shows the shape and dimensions of test piece.

Fig. 38. Izod impact test piece

/R0.25±0.025

Unit: mm

Notch Striking direction 12.7 ± 0.05 Parallel to R0.25±0.025 laminae 31.75±0.4 31.75±04 Perpendicular 63.5±0.6 to laminae

The width of test piece b shall be 12.7 mm as a standard, but may be reduced to the minimum of 2.0 mm depending on the conditions of the material under test.

The notch region (hereafter referred to as "V-notch") is formed by machining. The bottom of V-notch shall be finished smooth and be free from scars which may adversely affect the test. The angle included by the symmetrical planes of the V-notch and the longitudinal axis of test piece shall be $90 \pm 2^{\circ}$.

In the case of making the test for Izod impact strength (parallel to laminae), machine V-notch at right angles to the laminae. If the thickness of material under test is larger than 12.7 mm, machine either one face to 12.7 mm and machine V-notch.

If the thickness of material under test is less than 12.7 mm, direct the width of test piece "b" in the direction of thickness of base material.

In the case of making the test for Izod impact strength (perpendicular to laminae), machine V-notch in parallel with laminae (the machined surface, with the material of which one side is machined flat). Materials not more than 12 mm in thickness are exempted from the test.

5.21.3 Procedure Measure the width and thickness of test piece at the notch accurate to 0.01 mm, measure the dimensions of rectangular parallelepiped to the nearest 0.02 mm and confirm that each side is parallel to the opposite.

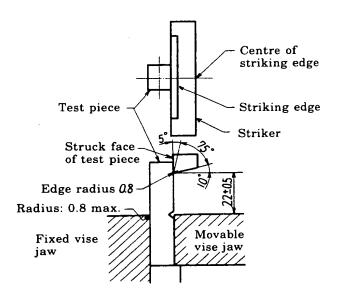
Confirm that the testing machine is installed horizontal in both fore-and-aft and lateral directions.

Set the test piece, as shown in Fig. 39, with the centre line of the notch in the same plane as the top face of vise, correctly align the centre of width with the centre of striking edge, and have the test piece gripped in the vise, confirming their mutual uniform contact.

Adjust the fastening pressure so that the fractured face of test piece is nearly parallel to the top face of vise.

Fig. 39. Setting Izod impact test piece

Unit: mm



Confirm that the freely hanging striking edge touches the struck face uniformly.

Hold up the pendulum and pointer in the starting position, next quickly release the pendulum and let it strike the test piece at a speed corresponding to the linear velocity of 3.3 m/s at the instant of impact. Read the excess swinging up angle β when the test piece has been broken by one impact, and determine the energy corresponding to β from the energy table.

5.21.4 Calculation Calculate Izod impact strength by the following equation:

$$A_{K1} = \frac{E}{b}$$

where, A_{K1} : Izod impact strength (J/m) {kgf·cm/cm}

E: energy when test piece has been broken by one

impact (J) {kgf·cm}

b: width of test piece at notch (cm)

5.22 Bonding strength (laminated sheets)

5.22.1 Apparatus

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- (1) Testing machine A testing machine which can be fitted with a steel ball of 10 mm diameter on its crosshead, and operated at a constant rate of crosshead motion. In this case, the allowable error in the load measuring system shall not exceed ± 1 % of the standard load of the material testing machine, and the load at cleaving shall be equivalent to not less than 15 % but not more than 85 % of its capacity.
- (2) <u>Micrometer calliper</u> The micrometer calliper for external measurement specified in JIS B 7502 or one with an accuracy at least equivalent.
- (3) Vernier calliper The vernier calliper giving the minimum reading value 0.02 mm specified in JIS B 7507 or one with an accuracy at least equivalent.
- 5.22.2 Test piece Use a test piece cut from the laminated sheet into the shape and dimensions shown in Fig. 40. In this case, if the thickness of the material is over 13 mm, shave off both sides equally to make the thickness 13 mm.

Fig. 40. Cleaving test method

Load
Steel ball
Test
piece
12.5
12.5
13±0.1

5.22.3 Procedure Measure the thickness of test piece with the external micrometer calliper accurate to 0.01 mm. Measure the height and width with the vernier calliper to the nearest 0.02 mm.

As shown in Fig. 40, centre the test piece in the material testing machine, and press it by means of a steel ball of 10 mm diameter, measure the load required to cleave the test piece to the nearest 10 N (1 kgf) and take this as the bonding strength (N) {kgf}.

Laminated sheets thinner than 13 mm are exempted from the test.

- 5.23 Appearance after heating
- 5.23.1 Moulding material To be tested as follows:
- (1) Apparatus
- (1.1) Thermostatic oven An oven capable of regulating the temperature within + 2°C tolerance over 100°C to 250°C.
- (1.2) Thermometer A thermometer graduated up to 360°C in 1°C divisions.
- (2) Test piece Use a test piece moulded into a disc of 50 ± 1 mm diameter and 3 ± 0.2 mm thickness.
- (3) Procedure Hang the test piece near the thermometer in the thermostatic oven maintained at the specified temperature for the kind of material, take it out after 2 h⁺ 6 min and examine it for remarkable changes such as cracks and blisters.
- 5.23.2 Laminated sheets To be tested as follows:
- (1) Apparatus To conform to 5.23.1 (1).
- (2) Test piece Use a test piece cut from the laminated sheet to a length and width of 50 ± 1 mm each with the full thickness.

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(3) Procedure To comply with 5.23.1 (3). But, cloth base laminated sheets of over 40 mm thickness, and asbestos base laminated sheets, glass fabric base laminated sheets and glass mat base laminated sheets of over 20 mm thickness are exempted from the test.

5.23.3 Laminated rods To be tested as follows:

- (1) Apparatus To conform to 5.23.1 (1).
- (2) Test piece Use a test piece cut from the laminated rod at right angles to the axis to a length of 50 ± 1 mm with the full diameter.
- (3) Procedure To comply with 5.23.1 (3). But, paper base laminated rods over 25 mm in diameter and cloth base laminated rods over 40 mm in diameter are exempted from the test.

5.23.4 Laminated tubes To be tested as follows:

- (1) Apparatus To conform to 5.23.1 (1).
- (2) Test piece Use a test piece cut from the laminated tube at right angles to the axis to a length of 50 ± 1 mm with the full diameter. However, with a laminated tube which can hardly be tested due to its large outside diameter a segment suitably divided from a tube section cut from the sample may be used.
- (3) Procedure To comply with 5.23.1 (3). However, paper base laminated tubes over 12.5 mm in wall thickness, cloth base laminated tubes over 20 mm in wall thickness and glass fabric base laminated tubes over 10 mm in wall thickness are exempted from the test.

5.24 Burning resistance

5.24.1 Method A To be conducted as follows:

(1) Apparatus

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- (1.1) Experimental stand A experimental stand provided with two clamps.
- (1.2) Burner and support Bunsen burner with 8.5 mm to 11.5 mm barrel diameter which uses town gas or LPG for fuel, and a support which can hold the burner inclined at 30° to the vertical. Measure beforehand the vertical height of the tip of flame above the bottom face of the support. The flame shall be a stable blue one with a height of about 25 mm.
- (1.3) Wire gauze A wire gauze equivalent to 850 µm of JIS Z 8801, having a length and width of approximately 100 mm each.
- (1.4) Stopwatch A stopwatch graduated in 0.2 s divisions.
- (2) $\frac{\text{Test piece}}{\text{into a size}}$ In the case of moulding material, use a test piece moulded into a size of approximately 127 mm length by 12.7 \pm 0.5 mm width and thickness.

In the case of laminated sheet use a test piece smoothly cut from the sample to a size of 127 mm length by $12.7^{+0.5}_{0}$ mm width, with the full thickness.

In every case, it shall be marked with gauge marks at 25 mm and 100 mm from one end (free end).

(3) Procedure In a room where no draught is sensible, as shown in Fig. 41, clamp the test piece at one end in the stand with its longitudinal axis horizontal, its transverse axis inclined at 45° to the horizontal, and its lower edge on the level of the tip of flame.

Place the burner 30° inclined to the vertical so that the tip of blue flame touches the free end of test piece for 30 s, then remove the flame, starting the stopwatch simultaneously.

Keep away the flame at least 450 mm from the test piece, stop the watch when the flame goes out and read the time in seconds to determine burning time. In this case, if the test piece continues to flame for 180 s or over, blow out the flame and define the material as combustible.

After extinction, measure the burnt length along the lower edge of test piece in millimeters (mm).

If the burnt length is not more than 25 mm, define the material as incombustible, and if over 25 mm but not more than 100 mm, as self-extinguishing.

Fig. 41. Burning resistance test apparatus

Test 100 piece Gauge mark

Free end

Wire gauze

Stand

Support

5.24.2 Method B To be conducted as follows:

- (1) Apparatus The apparatus shall be as follows. Fig. 42 shows the vertical burning test apparatus.
- (1.1) A draughtless chamber, enclosure, experimental hood
- (1.2) Bunsen burner (barrel length of 101.6 mm, inside diameter of 9.5 mm)
- (1.3) A ring stand provided with a clamp
- (1.4) Industrial methane or a cylinder held natural gas of 37 MJ/m³ (approximately 9000 kcal/m³) (provided with a flow rate meter, regulator)
- (1.5) Dry surgery cotton
- (1.6) A stopwatch or timer
- (1.7) A desiccator (containing dry calcium chloride or silica gel)
- (1.8) A hot air type thermostatic oven capable of maintaining the temperature at $70 \pm 1^{\circ}C$
- (2) Test piece In the case of moulding material, use a moulded test piece 127 mm in length by $12.7^{+0.5}_{0}$ mm in width by not less than 0.8 mm in thickness. In the case of laminated sheet, use a test piece smoothly cut from a sample into a size 127 mm in length by $12.7^{+0.5}_{0}$ mm in width, with the full thickness.

In both cases, the corner radius of test piece R shall not exceed 1.27 mm.

Unit: mm

- (3) Preconditioning Test pieces shall be preconditioned on the following two:
- (3.1) The condition as furnished.

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- (3.2) Heating at $70 \pm 1^{\circ}$ C temperature for 168 h and allowing to stand in the desiccator for not less than 4 h.
- (4) Number of test pieces To be 5 for each precondition as shown in (3).
- (5) Procedure Adjust the burner to produce a blue flame about 19 mm in height, and apply the flame to the centre of lower end of test piece hung by the clamp with its longitudinal axis vertical for 10 s (the relative positions of the test piece to the burner and the surgery cotton laid below the test piece shall be as shown in Fig. 42). After the application of flame, withdraw the burner from the test piece not less than 152 mm and measure the time of flaming.

As soon as the flaming has ceased, apply the flame again to the same point of test piece for 10 s, then withdraw it not less than 152 mm and measure the time of flaming and time of glowing.

Note the thickness of test piece in addition to the results.

Test piece

12.7

Burner

51 × 51

Fig. 42. Apparatus for vertical burning test

(6) Requirements for test results Classify the materials conforming to the following requirements under the conditions of both 5.24.2 (3.1) and (3.2), into Class V-0 and Class V-1, respectively.

Surgery cotton

	Test condition	Burning resistance		
		Class V-0	Class V-1	
(1)	Time of flaming after withdrawing flame	10 s max.	30 s max.	
(2)	Total time of flaming when a set of 5 test pieces are applied with flame 10 times	50 s max.	250 s max.	
(3)	Time of glowing after withdrawing flame of second application	30 s max.	60 s max.	
(4)	Occurrence of dripping of material which can ignite surgery cotton 305 mm below test piece	Not allowed	Not allowed	
(5)	Occurrence of flaming or glowing reaching clamp	Not allowed	Not allowed	
(6)	If one among a set of five fails or the total time of flaming for the five in a set falls within the range shown to the right, make a retest on another set of five and the results shall meet all the requirements of (1), (2), (3).	51 s to 55 s	251 s to 255 s	

5.24.3 Method C

- (1) Apparatus The apparatus shall have the constitution as shown in Fig. 43.
- (1.1) A draughtless chamber, enclosure, experimental hood
- (1.2) Bunsen burner (barrel length of 101.6 mm, inside diameter of 9.5 mm)
- (1.3) A ring stand provided with a clamp
- (1.4) Industrial methane or a cylinder held natural gas of 37 MJ/m³ (approximately 9000 kcal/m³) (provided with a flow rate meter, regulator)
- (1.5) Wire gauze [840 µm (20 mesh), 127 mm square]
- (1.6) A stopwatch or timer
- (2) Test piece In the case of moulding material, use a moulded test piece 127 mm in length by $12.7^{+0.5}_{0} \text{ mm}$ in width by not less than 0.8 mm in thickness. In the case of laminated sheet, use a test piece smoothly cut from a sample into a size 127 mm in length by $12.7^{+0.5}_{0} \text{ mm}$ in width with the full thickness. In both cases, the corner radius of test piece R shall not exceed 1.27 mm.
- (3) Preconditioning To be the condition as furnished.
- (4) Number of test pieces To be three.
- (5) Procedure Gauge marks shall be drawn across the test pieces at 25.4 mm and 101.6 mm from one end.

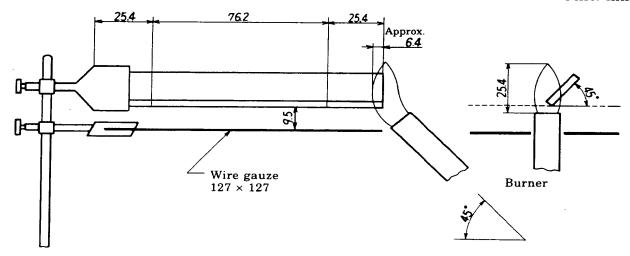
Light the burner at a distance from the test piece and adjust the height of blue flame to 25.4 mm. Apply the testing flame for 30 s, then withdraw it. If the test piece has burnt to the 25.4 mm gauge mark within 30 s of the application of testing flame, withdraw the flame at that instant. If the test piece continues to burn, measure the time of the flame from passing the 25.4 mm gauge mark to reaching the 101.6 mm one and determine the burning speed.

Note the thickness of test piece in addition to the results.

- (6) Requirements for test results Classify the material conforming to the following requirements into Class HB.
- (6.1) In the case the material does not conform to Class V-0 and Class V-1 specified in 5.24.2 (6), the flame shall extinguish before it reaches the 101.6 mm gauge mark, or the burning speed on 76 mm span shall be not more than 38.1 mm/min for the test piece 3.05 mm to 12.7 mm in thickness or not more than 76.2 mm/min for that less than 3.05 mm in thickness.
- (6.2) In the case where one of the three test pieces in a set does not conform to 5.24.3 (6.1), another set of three shall all conform to 5.24.3 (6.1).

Fig. 43. Apparatus for horizontal burning test

Unit: mm



- 5.25 Thermal expansion
- 5.25.1 Moulding material To be tested as follows:
- (1) Apparatus
- (1.1) Suitable heater A heater capable of raising the temperature from room temperature to about 80°C in about 1 h.
- (1.2) Micrometer calliper The micrometer calliper for external measurement specified in JIS B 7502 or one with an accuracy at least equivalent.
- (1.3) Thermometer A thermometer graduated in 1°C divisions up to 100°C.
- (2) Test piece Use a test piece moulded to a length of 120 mm by width and thickness of about 10 mm each.
- (3) Procedure Put the test piece in the heater and measure its expansion with the external micrometer calliper to the nearest 0.01 mm, raising the temperature from room temperature to about 80°C in about 1 h.
- (4) <u>Calculation</u> Calculate the rate of thermal expansion for 1°C temperature difference by the following equation:

$$\alpha = \frac{l}{L(t_2 - t_1)}$$

where, α : rate of thermal expansion for 1°C temperature difference

L: length of test piece along expanding axis at start of test (mm)

l: expansion (mm)

t₂: temperature in heater when expansion is measured (°C)

 t_1 : room temperature at start of test (°C)

5.25.2 Laminated sheets To be tested as follows:

Laminated sheets less than 10 mm thick are exempted from the test.

- (1) Apparatus To conform to 5.25.1 (1).
- (2) Test piece test for the test for thermal expansion vertical to laminae, use a test piece cut from the laminated sheet to a length and width of about 10 mm each, with the full thickness. For the test for thermal expansion parallel to laminae, use a test piece cut from the laminated sheet to a length of about 120 mm by width and thickness of about 10 mm each.
- (3) Procedure Conduct the test in accordance with 5.25.1 (3).
- (4) Calculation Make the calculation in accordance with 5.25.1 (4).

5.26 Water absorption

5.26.1 Moulding material

- (1) Apparatus
- (1.1) Balance A balance with reciprocal sensibility 1 mg.
- (1.2) Weighing bottle A weighing bottle with a size capable of accepting the test piece, and of being tightly plugged.
- (1.3) Thermostatic tank water at 23 ± 0.5°C. A thermostatic tank capable of maintaining distilled
- (1.4) Thermostatic oven $\frac{1}{2}$ A thermostatic oven capable of maintaining the temperature at 50 ± 2 °C.
- (1.5) Desiccator A desiccator containing dry calcium chloride or silica gel.
- (1.6) Glass vessel for water absorption provided with cover a glass vessel capable of accepting the test piece.
- (1.7) Thermometer A thermometer graduated up to 50°C in 0.1°C divisions, and another one up to 100°C in 1°C divisions.
- (2) Test piece Use a test piece moulded in the form of a disc 50 ± 1 mm in diameter and 3 ± 0.2 mm in thickness.
- (3) Preconditioning Dry the test piece in the thermostatic oven maintained at 50 ± 2°C for 24 ± 1 h. In that, lay the test piece on the filter paper laid on an asbestos plate about 10 mm thick.

(4) Procedure Cool the test piece after preconditioning, in the desiccator to $20 \pm 10^{\circ}$ C and measure its mass accurate to 1 mg.

Next, immerse it in the distilled water at $23 \pm 0.5^{\circ}C$ contained in the vessel for water absorption for 24 ± 1 h, take it out, wipe it with dry clean gauze or the like, brush away the dust on the surface with a feather or writing brush, put it in the weighing bottle within 1 min and measure the mass after absorption accurate to 1 mg. In the above, take care to avoid mutual contact of the test pieces during immersion.

(5) Calculation Calculate water absorption by the following equation:

$$A = \frac{W_2 - W_1}{W_1} \times 100$$

where, A: water absorption (%)

 W_1 : mass of test piece before water absorption (g)

 W_2 : mass of test piece after water absorption (g)

5.26.2 Laminated sheets

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(1) Apparatus To conform to 5.26.1 (1).

- (2) Test piece Use a test piece cut from the laminated sheet to a length and width of 50 ± 1 mm each, with the full thickness. Finish the cut faces smooth with the abrasive paper specified in JIS R 6252 or the like.
- (3) Preconditioning To be preconducted in accordance with 5.26.1 (3).
- (4) Procedure To be conducted in accordance with 5.26.1 (4).
- (5) <u>Calculation</u> To be done in accordance with 5.26.1 (5).

5.26.3 Laminated rods To be tested as follows:

- (1) Apparatus To conform to 5.26.1 (1).
- (2) Test piece Use a test piece cut from the laminated rod at right angles to the axis to a length of 50 ± 1 mm with the full diameter. Finish the cut faces smooth with the abrasive paper specified in JIS R 6252 or the like.
- (3) <u>Preconditioning</u> To be preconducted in accordance with 5.26.1 (3).
- (4) Procedure To be conducted in accordance with 5.26.1 (4). But laminated rods less than 6 mm in diameter are exempted from the test.
- (5) <u>Calculation</u> To be done in accordance with 5.26.1 (5).

5.26.4 <u>Laminated tubes</u> To be tested as follows:

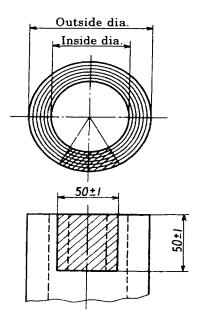
- (1) Apparatus To conform to 5.26.1 (1).
- (2) Test piece Use a test piece cut from the laminated tube at right angles to the axis to a length of 50 ± 1 mm with the full diameter. However, for laminated tubes not less than 75 mm in inside diameter, use one, as shown in Fig. 44, cut from within its wall to a length and width of 50 ± 1 mm each. Take the test piece from the part of laminated tube where the end face is showing no cracks or crevices and finish its cut faces smooth with the abrasive paper specified in JIS R 6252 or the like.

- (3) Preconditioning To be conducted in accordance with 5.26.1 (3).
- (4) Procedure To be conducted in accordance with 5.26.1 (4). But laminated tubes less than 1 mm in wall thickness are exempted from the test.
- (5) Calculation To be done in accordance with 5.26.1 (5).

Fig. 44. Test piece for water absorption of laminated tube

Unit: mm

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- 5.27 Boiling water absorption (moulding material)
- 5.27.1 Apparatus Use the following:
- (1.1) Balance A balance with reciprocal sensibility 1 mg or better.
- (1.2) Weighing bottle A weighing bottle with a size capable of accepting the test piece, and of being tightly plugged.
- (1.3) Beaker A beaker with suitable capacity for boiling the test piece in it.
- (1.4) Thermostatic oven $\frac{\text{Thermostatic oven capable of adjusting the temperature at 50 <math>\pm$ 2°C.
- (1.5) Desiccator A desiccator containing dry calcium chloride or silica gel.
- (1.6) Thermometer A thermometer graduated up to 100°C in 1°C divisions.
- 5.27.2 Test piece Use a test piece moulded in the form of a disc 50 ± 1 mm in diameter and 3 ± 0.2 mm in thickness
- 5.27.3 Preconditioning Dry the test piece in the thermostatic oven maintained at $50 \pm 2^{\circ}$ C for 24 ± 1 h. In that, lay the test piece on the filter paper laid on an asbestos plate about 10 mm thick.

5.27.4 Procedure Cool the test piece after conditioning, in the desiccator to $20 \pm 10^{\circ} \text{C}$ and measure its mass accurate to 1 mg. Next, after boiling it in distilled water for 1 h, take it out and cool it in running fresh water at $20 \pm 10^{\circ} \text{C}$ for 30 min, wipe it with dry clean gauze or the like, brush away the dust on the surface with a feather or writing brush, put it in the weighing bottle within 1 min and measure the mass after water absorption accurate to 1 mg. In the above, take care to avoid mutual contact of the test pieces during boiling.

5.27.5 <u>Calculation</u> Calculate boiling water absorption by the following equation:

$$A = \frac{W_2 - W_1}{W_1} \times 100$$

where, A: boiling water absorption (%)

 W_1 : mass of test piece before boiling (g)

 W_2 : mass of test piece after boiling (g)

5.28 Specific gravity

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5.28.1 Apparatus Use those as follows:

- (1) Balance A balance with reciprocal sensibility 1 mg or better.
- (2) Thermometer A thermometer graduated up to 100°C in 1°C divisions.
- (3) Beaker and support for it suitable support for it. The beaker specified in JIS R 3503 and a
- (4) Thermostatic apparatus $\frac{\text{Thermostatic apparatus capable of regulating}}{\text{the temperature at } 23 \pm 2^{\circ}\text{C}}$.
- 5.28.2 <u>Test piece</u> Use a test piece made from the laminate (sheet, rod or tube) or moulding. Make the surfaces of test piece smooth by a suitable method to avoid adhesion of bubbles when the test piece is immersed in liquid. The size of test piece shall be suitable to the beaker used in measurement and 1 g to 5 g of the material shall be taken.
- 5.28.3 Procedure Measure the mass of test piece accurate to 1 mg in $23 \pm 2^{\circ}$ C air. Take newly distilled water or distilled water having been boiled and deaerated prior to measurement as the immersing liquid, pour it in the beaker and maintain it at $23 \pm 2^{\circ}$ C, and mount the beaker on the support placed avoiding contact with the pan of balance.

Immerse the test piece hung by a fine metal wire in the immersion liquid and hook the other end of metal wire on the balance. Measure their mass accurate to 1 mg.

Next, remove the test piece from the wire and measure the mass of the metal wire alone in the same condition as the above accurate to 1 mg.

5.28.4 Calculation Calculate specific gravity by the following equation:

$$S = \frac{W}{W - W_1 + w}$$

where, S: specific gravity

W: mass of test piece in air (g)

 W_1 : mass when the test piece is suspended in

immersion liquid by metal wire (g)

w: mass when metal wire alone is suspended in

immersion liquid (g)

5.29 Resistance to acetone (for phenolic resin only)

5.29.1 Moulding material To be tested as follows:

- (1) Apparatus
- (1.1) The flask specified in JIS R 3503 with suitable size for holding the test piece, and a cooler.
- (1.2) A water bath capable of accepting the flask and maintaining it at a suitable temperature.
- (2) Test piece Use a test piece moulded to a length, width and height of about 20 mm each.
- (3) Testing liquid Use the acetone specified in JIS K 8034.
- (4) Procedure Put the test piece and testing liquid in the flask provided with the cooler, immerse the flask in the water bath, boil the testing liquid for 30 min, take out the test piece and immediately examine it for crazing, blister or other remarkable change in appearance.

5.29.2 Laminated sheets To be tested as follows:

- (1) Apparatus To conform to 5.29.1 (1).
- (2) Test piece Use a test piece cut from the laminated sheet to a suitable shape and dimensions with the full thickness.
- (3) Testing liquid To conform to 5.29.1 (3).
- (4) Procedure To be conducted in accordance with 5.29.1 (4).

5.29.3 Laminated rods To be tested as follows:

- (1) Apparatus Use the beaker specified in JIS R 3503 with a suitable size for holding the test piece.
- (2) Test piece Use a test piece cut from the laminated rod to a suitable shape and dimensions.

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(3) Testing liquid To conform to 5.29.1 (3).

- (4) Procedure At 23 ± 10°C room temperature, put the test piece and testing liquid in the beaker, allow them to stand for 2 h, then take out the test piece and examine it for the extent of change in appearance. In the above, it is preferable to avoid volatilization of testing liquid by putting a cover on the beaker during testing.
- 5.29.4 Laminated tubes To be tested as follows:
- (1) Apparatus To conform to 5.29.3 (1).
- (2) Test piece Use a test piece cut from the laminated tube to a suitable shape and dimensions.
- (3) Testing liquid To conform to 5.29.1 (3).
- (4) Procedure To be conducted in accordance with 5.29.3 (4).
- 5.30 Resistance to boiling water (moulding material)
- 5.30.1 Apparatus
- (1) Beaker The beaker specified in JIS R 3503 with a size capable of holding and perfectly immersing the test piece.
- (2) Glass support A support about 10 mm high for keeping the test piece off the bottom and side of beaker.
- 5.30.2 Test piece Use a test piece moulded to a diameter of 50 ± 1 mm and thickness of 3 ± 0.2 mm.
- 5.30.3 Preconditioning To be in the condition as furnished.
- 5.30.4 Procedure Place the glass support in the beaker, pour in distilled water and boil it, lay the test piece horizontal on the support, boil it for 15 min, take it out and examine it for the extent of change in appearance and the boiling water for coloration.
- 5.31 Resistance to sulfuric acid (for melamine resin moulding material only)
- 5.31.1 Apparatus

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- (1) Beaker The beaker specified in JIS R 3503 with a suitable size for holding the test piece.
- (2) Glass support test piece. A support about 10 mm high capable of supporting the
- 5.31.2 Test piece Use a test piece moulded to a diameter of 50 ± 1 mm and thickness of 3 ± 0.2 mm.
- 5.31.3 Testing liquid Use the liquid of sulfuric acid specified in JIS K 8951 diluted with distilled water to (0.8 ± 0.05) % concentration.

5.31.4 Procedure Place the glass support in the beaker, pour in 300 ml of testing liquid and boil it, lay the test piece horizontal on the support, boil it for 10 min, take it out and examine it for the extent of change in appearance such as change in gloss, cracking, blushing and discoloration.

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5.32 Chemical resistance (moulding material)

5.32.1 Apparatus

- (1) Balance A balance with reciprocal sensibility 1 mg or better.
- (2) <u>Container</u> A glass wide-mouthed bottle or other suitable container, with a size capable of holding the testing liquid and completely immersing the test piece.

As a general rule, use one vessel for each test piece. Test pieces from the same sample may be put in the same container in numbers.

- (3) Thermostatic oven perature at $50 \pm 2^{\circ}C$. A thermostatic oven capable of adjusting the temperature at $50 \pm 2^{\circ}C$.
- (4) Weighing bottle The weighing bottle specified in JIS R 3503, with a size capable of accepting the test piece and being tightly plugged.
- (5) Thermostatic apparatus A thermostatic apparatus capable of maintaining the temperature of immersion testing container within ± 2°C of the specified temperature.
- 5.32.2 Test piece Use a test piece moulded into a disc 50 ± 1 mm in diameter and 3 ± 0.2 mm in thickness.
- 5.32.3 <u>Preconditioning</u> Dry the test piece in the thermostatic oven maintained at $50 \pm 2^{\circ}$ C for 24 ± 1 h. In that, lay the test piece on the filter paper laid on an asbestos plate about 10 mm thick.
- 5.32.4 <u>Testing liquid</u> The chemical reagents used for the testing liquid shall be those specified in Japanese Industrial Standards. The chemical reagents not specified in Japanese Industrial Standards shall be specified for their quality by agreement between the parties concerned.

The quantity of testing liquid used shall as a rule be 8 ± 2 ml for 1 cm^2 of surface area of test piece.

Table 8 shows examples of testing liquid used for evaluating the chemical resistance of test piece.

Table 8. Examples of testing liquid

Reagent	Concentration %	Reagent	Concentration %
Distilled water		Hydrochloric acid	10
Sulfuric acid	10	Hydrochloric acid	35
Sulfuric acid	30	Nitric acid	10
Sulfuric acid	80	Nitric acid	40
Sulfuric acid	98	Nitric acid	60

Reagent	Concentration %	Reagent	Concentration %
Glacial acetic acid	99 to 100	Ethyl alcohol	50v/v
Acetic acid	5	Ethyl alcohol	95v/v
Citric acid	10	Acetone	95v/v
Sodium hydroxide	10	Ethyl acetate	95v/v
Sodium hydroxide	40	Carbon tetrachloride	95v/v
Sodium carbonate	2	Trichloroethylene	95v/v
Sodium carbonate	20	Benzene	95v/v
Aqueous ammonia	10	Gasoline	
Aqueous ammonia	28	Kerosene	
Sodium chloride	10	Animal oil, vegetable oil	
Methyl alcohol	95v/v	· -	

Table 8. (continued)

5.32.5 Procedure Measure the mass of test piece after preconditioning accurate to 1 mg. Next, immerse it in the testing liquid maintained at a constant temperature. Keep the test piece not to closely contact the container wall and gently sway the container every 24 h to stir the testing liquid. Take out the test piece after a fixed period of time, quickly wash it with distilled water or suitable liquid, wipe it with dry clean gauze or the like, immediately put it into the weighing bottle and measure the mass accurate to 1 mg to determine the change in mass.

For determining the quantity of extract by a volatile solvent, after immersion of the test piece, exsiccate it again at $50 \pm 2^{\circ}C$ for 24 ± 1 h, then measure its mass accurate to 1 mg and make calculation therefrom.

Observe the appearance of test piece after testing, for loss of gloss, discoloration, haze, crazing, cracking, swelling, warping, decomposition, dissolution, etc.

Note the name and concentration of testing liquid and the testing conditions.

5.32.6 <u>Calculation</u> Calculate the changing rate of mass by the following equation:

$$\Delta W = \frac{W_2 - W_1}{W_1} \times 100$$

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where, ΔW : changing rate of mass (%)

 W_1 : mass of test piece after conditioning (g)

 W_2 : mass of test piece after immersion (g)

Calculate the ratio of the mass of extracted substance to that of test piece by the following equation:

$$\Delta W_e = \frac{W_1 - W_3}{W_1} \times 100$$

where, ΔW_e : ratio of mass of extracted substance to that of

test piece (%)

 W_1 : mass of test piece after conditioning (g)

 W_3 : mass of test piece after immersion and

exsiccation (g)

5.33 Free water content (moulding material) To be tested as follows:

- (1) Apparatus
- (1.1) Balance A balance with a weighing capacity of 100 g to 200 g and reciprocal sensibility 1 mg.
- (1.2) Weighing bottle in JIS R 3503. The flat weighing bottle of 50 mm \times 30 mm specified
- (1.3) Desiccator A desiccator containing dry calcium chloride or silica gel.
- (2) Procedure Weigh the clean weighing bottle having been exsiccated beforehand to the nearest 1 mg. Put 5 ± 0.1 g of sample in the weighing bottle, spread it to a uniform thickness as far as practicable, apply the plug and measure its mass accurately.

Next, put it in the desiccator, dry it for 96 + 1 h with the plug taken off, immediately plug and weigh it accurately.

(3) <u>Calculation</u> Calculate the free water content by the following equation to the first decimal place:

$$W = \frac{w_1 - w_2}{w_1} \times 100$$

where, W: free water content (%)

 w_1 : mass of sample before exsiccation (g)

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 w_2 : mass of sample after exsiccation (g)

- 5.34 Flexural strength under heat (laminated sheets)
- 5.34.1 Apparatus The apparatus shall conform to 5.17.1 (1). In this case, the material testing machine shall provide an thermo-device which can maintain the test piece holding parts at a fixed temperature utilizing the air as heat-transfer medium.
- 5.34.2 Test piece To conform to 5.17.2 (2).
- 5.34.3 Procedure Measure the height h (mm) and width W (mm) of the test piece accurate to 0.01 mm with an external micrometer calliper, and raise the test piece holding parts of apparatus to $150 \pm 2^{\circ}$ C. Next, support the test piece with a span of $16h \pm 0.5$ mm, keep it in the state for 60^{+6} min, then without changing the condition apply a load at the centre of test piece by the compressing nose as shown in Fig. 24, and measure the load at the break of test piece to the nearest 1 N $\{0.1 \text{ kgf}\}$. If the test piece has broken outside the central portion of its trisections, discard the measurement and make a retest.

Calculate loading speed by the following equation:

$$V = \frac{h}{2} \pm 0.2$$

where, V: loading speed (mm/min)

h: height of test piece (mm)

5.34.4 <u>Calculation</u> Calculate the bending strength under heat and the rate of retaining bending strength under heat by the following equation:

(1) Flexural strength under heat

$$\sigma_{hfB} = \frac{3PL_V}{2Wh^2}$$

where, $\sigma_{h/B}$: flexural strength under heat (MPa) {kgf/mm²}

P: load at break of test piece (N) {kgf}

 L_v : support span (mm)

W: width of test piece (mm)

h: thickness of test piece (mm)

(2) Rate of retaining flexural strength under heat

$$K = \frac{\sigma_{h/B}}{\sigma_{/B}} \times 100$$

where, K: rate of retaining flexural strength under heat (%)

 $\sigma_{\!f\!B}$: flexural strength at ordinary temperature (MPa)

{kgf/mm²}

 $\sigma_{h/B}$: flexural strength under heat (MPa) {kgf/mm²}

5.35 Deflection temperature under load

5.35.1 Moulding material To be tested as follows:

(1) Apparatus

(1.1) It shall consist of a loading rod fitted with weight pan, supports for test piece, a dial gauge, weights, a metal frame for supporting these, a heating bath and a thermometer, as shown in Fig. 45.

Weight Weight pan

Thermometer

Dial gauge

Heating

Fig. 45. Apparatus for load-deflection test

Heating bath

Frame

Loading rod

Loading nose

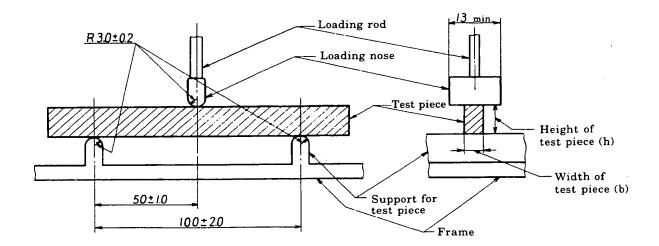
Test piece

(1.1.1) The supports for test piece shall be metal supports which can support a

(1.1.1) The supports for test piece shall be metal supports which can support a test piece horizontal with a span of 100 ± 2.0 mm as shown in Fig. 46. The loading rod shall be supported by the frame to permit vertical motion, and the loading nose at its end shall have a curved surface 3.0 ± 0.2 mm in radius and not less than 13 mm in width. The loading rod, frame and supports for test piece shall be made of the same material with little coefficient of thermal expansion.

Fig. 46. Method for supporting test piece

Unit: mm



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(1.1.2) The weight pan shall be attached to the loading rod and of a construction that may not cause eccentricity when loaded with weight. Weights used shall be of a suitable amount to apply a load (5) producing a bending stress of 1.80 N/mm² {18.5 kgf/cm²} in the test piece.

They may be of an amount to produce a bending stress of 0.45 MPa {4.6 kgf/cm²} subject to agreement between the parties concerned.

The load shall be calculated by the following equation:

$$P = \frac{2\sigma bh^2}{3l}$$

where,

 $P : load(N) \{kgf\}$

 σ : bending stress produced in test piece (MPa) {kgf/

 cm^2

b: width of test piece (cm)

h: height of test piece (cm)

l: support span for test piece (cm)

Note (5) The actual load applied on the test piece shall be within \pm 2.5 % of calculated load which is the sum of the weights, loading rod with the weight pan and the thrust by the dial gauge. Pay attention to the sense of the thrust by the dial gauge, and the force shall be added or subtracted according to the arrangement.

(1.1.3) The heating bath shall consist of a bath to hold the heat-transfer $medium(^6)$, stirrer and heating device which can raise the temperature of heat-transfer medium at a constant rate of 2 ± 0.2 °C/min.

A heat-transfer medium which will not affect the quality of test piece shall be used.

Note $^{(6)}$ It is preferable to use as the heat-transfer medium a silicone oil having the dimethylcyloxane or diphenylcyloxane constitution and a viscosity of 1 to 5×10^{-4} m²/s {100 cSt to 500 cSt} at 25°C.

(1.2) The dial gauge used for measuring the deflection of test piece shall be of the scale interval 0.01 mm specified in JIS B 7503 or one with an accuracy at least equivalent.

The vernier calliper used for measuring the dimensions of test piece and span of supports for test piece shall be of the minimum reading value 0.05 mm specified in JIS B 7507 or one with an accuracy at least equivalent.

The thermometer shall be a mercury thermometer with suitable temperature measuring range and graduated in $0.5^{\circ}\mathrm{C}$ or less.

Further, the thermometer to be used shall previously be corrected and verified that the error is within 0.5°C. Any electrical thermometer may be used, provided its accuracy is equivalent to the mercury thermometer.

(1.3) Correction of testing apparatus Prior to testing, correction shall be made for thermal expansion, thermometer and load.

- (1.3.1) Correction for thermal expansion Correction for thermal expansion is determined by using a test piece made of borosilicate glass (not less than 3 mm wide × 12.7 ± 0.2 mm deep × not less than 110 mm long) as the standard test piece. If the reading on the dial gauge by the thermal expansion within the range of testing temperatures exceeds 0.01 mm, the measurement shall be corrected.
- (1.3.2) Correction of thermometer The thermometer shall be corrected by the difference between the standard and used thermometer within the range of testing temperatures.
- (1.3.3) Correction of load Since the sum of loading rod (including loading nose), weight pan and thrust of dial gauge may exceed the nominal value due to frictional resistance and others, it shall be calibrated after assembly of the apparatus and corrected with weights accordingly.
- (2) Test piece The test piece shall be 6.4 ± 0.2 mm in width by 12.7 ± 0.2 mm in height by not less than 110 mm in length. However, a width of 3.2 ± 0.2 mm or 12.7 ± 0.2 mm may be used, subject to agreement between the parties concerned.
- (3) Procedure Measure the width b and height h of the test piece to the nearest 0.1 mm, lay the test piece horizontal on the supports as shown in Fig. 46 and immerse it in the heat-transfer medium of room temperature (7). Next, put on a load determined to produce a bending stress of 1.80 MPa $\{18.5 \text{ kgf/cm}^2\}$ in the test piece for 5 min, and record the zero point of dial gauge. Raise the temperature of heat-transfer medium at a rate of 2 ± 0.2 °C/min, and measure the temperature of heat-transfer medium when the deflection of test piece reaches 0.26 mm to the first decimal place.

Place the mercury bulb of thermometer off but within 2 mm of the test piece.

Note (7) In immersing the test piece in the heat-transfer medium, adjust the distance between the surface of liquid and the surface of test piece becomes at least 35 mm.

5.35.2 Laminated sheets To be tested as follows:

- (1) Apparatus To conform to 5.35.1 (1).
- (2) Test piece
- (2.1) Make the test piece by cutting out a part of the laminated sheet by machining (8) to a length of not less than 110 mm by width of 6.4 ± 0.2 mm by height of 12.7 ± 0.2 mm. A width of 3.2 ± 0.2 mm or 12.7 ± 0.2 mm may be used, subject to agreement between the parties concerned.
 - Note (8) In machining, careful attention shall be paid to avoiding the change in quality by the heat arising from machining and others.
- (3) Procedure To be conducted in accordance with 5.35.1 (3). In that, the direction of application of load shall be parallel to laminae.

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Hardness testing methods for rubber, vulcanized or thermoplastic

JIS K 6253:1997

Introduction This Japanese Industrial Standard has been prepared on the basis of the 3rd edition of ISO 48, Rubber, vulcanized or thermoplastic—Determination of hardness (hardness between 10 IRHD and 100 IRHD) published in 1994, and the 1st edition of ISO 7619, Rubber—Determination of indentation hardness by means of pocket hardness meters published in 1986, without any modification in technical contents. However, "Type E of spring type (durometer hardness)" which is not specified in the corresponding International Standards are added in this Standard.

1 Scope This Japanese Industrial Standard specifies the testing methods to measure hardness of vulcanized rubber and thermoplastic rubber (hereafter referred to as "vulcanized rubber").

Remarks 1 The standards cited in this Standard are listed as follows.

JIS K 6200 Glossary of terms used in rubber industry

JIS K 6250 General rules of physical testing methods for rubber, vulcanized or thermoplastic

JIS Z 8401 Rules for rounding off of numerical values

2 The International Standards corresponding to this Standard are listed as follows.

ISO 48:1994 Rubber, vulcanized or thermoplastic—Determination of hardness (hardness between 10 IRHD and 100 IRHD)

ISO 7619: 1986 Rubber—Determination of indentation hardness by means of pocket hardness meters

- 3 The units and numerical values given in { } in this Standard are based on traditional units, and are appended for informative reference.
- 2 Definitions For the purposes of this Standard, the definitions given in JIS K 6200 and JIS K 6250, and the following definitions apply.
- (1) international rubber hardness degree Hardness which can be obtained through conversion into international rubber hardness degree (IRHD)(1) using the depth of indentation by a plunger when the plunger, with a ball-type lower end, is vertically impressed on the surface of a test piece with specified indenting force.

A hardness scale is chosen so that "0" represents the hardness of material having a Young's modulus of zero and "100" represents the hardness of a material of infinite Young's modulus, and the following conditions are fulfilled over most of normal range of hardness.

- (a) One international rubber hardness degree always represents approximately the same proportionate difference in the Young's modulus.
- (b) For highly elastic rubber, the scales of international rubber hardness degree and that of type A durometer are comparable.

Note (1) IRHD International Rubber Hardness Degree

- (2) durometer hardness The hardness given by the testing apparatus (durometer) which reads the indentation depth made by a specifically shaped indentor when it is impressed on the surface of a test piece via a spring.
- (3) **IRHD pocket hardness** The hardness given by a portable pocket testing apparatus (IRHD pocket hardness meter) by which international rubber hardness degree can be conveniently obtained owing to reading the indented depth made by an indentor, with a ball-type lower end, when it impressed on the surface of a test piece via a spring.
- (4) **standard hardness** The hardness obtained using the specified procedures on test pieces whose shape and dimensions satisfy the specifications, when carrying out each test.
- (5) **apparent hardness** The hardness obtained either using other procedures than the specified, or on the test piece whose shape and dimensions do not satisfy the specification, when carrying out each test.

3 Type of test

3.1 Outline of hardness test There are many types of testing methods for hardness test depending on the principle of hardness measurement, range of hardness measurement, kind of testing apparatus and so on, and they are classified into standard hardness and apparent hardness by shape or dimensions of a test piece. The outline of classifying is shown in Table 1.

Table 1 Outline of hardness tests

Principle of	Range of hardness measurement	Type of testing apparatus	Testing method	Test condition for standard hardness		
measurement				Shape	Thickness mm	Minimum distance from the edge of sample mm
Constant-	For high hardness (85 to 100 IRHD)	Normal size inter- national rubber hardness meter	H method	surfaces are smooth and parallel	8.0 min.	9.0
force type (international					10.0 max.	10.0
rubber hardness	For normal hardness (30 to 95 IRHD)	Normal size inter-	N		8.0 min.	9.0
degree)		national rubber hardness meter	method		10.0 max.	10.0
		Microsize inter- national rubber hardness meter	M method		1.5 min.	2.0
					2.5 max.	
	For low hardness (10 to 35 IRHD)	Normal size inter- national rubber	L method		10.0 min.	10.0
		hardness meter			15.0 max.	11.5
Spring type (durometer hardness)	For high hardness (A90 or more)	Type D durometer			6.0 or more	12.0
nardness)	For normal hardness (A10 to 90)	Type A duromete	r		6.0 or more	12.0
	For low hardness (A20 or less)	Type E durometer			10.0 or more	12.0
Spring type (IRHD pocket hardness)	For normal hardness (30 to 95 IRHD)	IRHD pocket hardness meter	P method		6.0 or more	12.0

- **3.2 Type of tests** The type of hardness tests for vulcanized rubber shall be classified as follows.
- (1) International rubber hardness test
 - (a) H method (normal size test for high hardness)
 - (b) N method (normal size test for normal hardness)
 - (c) M method (microsize test for normal hardness)
 - (d) L method (normal size test for low hardness)
- (2) Durometer hardness test
 - (a) Type D (test for high hardness)
 - (b) Type A (test for normal hardness)
 - (c) Type E (test for low hardness)
- (3) IRHD pocket hardness test
 - (a) P method (for normal hardness)
- 4 International rubber hardness test
- 4.1 Purpose This test shall be carried out to measure the international rubber hardness degree of vulcanized rubber.
- 4.2 Range of measurement The measuring range of this test is decided according to the thickness and hardness of a test piece for every testing method. The measuring range of each testing method is as follows.
- (1) **H method** Formal measuring range shall be for the test piece measuring 8.0 mm to 10.0 mm in thickness and with hardness of 85 IRHD to 100 IRHD. It is permissible to test the one with 4.0 mm or more thickness and with hardness of 85 IRHD to 100 IRHD.
- (2) N method Formal measuring range shall be for the test piece measuring 8.0 mm to 10.0 mm in thickness and with hardness of 35 IRHD to 85 IRHD. It is permissible to test the one with 4.0 mm or more thickness and with hardness of 30 IRHD to 95 IRHD(2).
- (3) **M method** Formal measuring range shall be for the test piece measuring 1.5 mm to 2.5 mm in thickness and with hardness of 35 IRHD to 85 IRHD. It is permissible to test the one with 1.0 mm to 4.0 mm thickness and with hardness of 30 IRHD to 95 IRHD(3).
- (4) L method Formal measuring range shall be for the test piece measuring 10.0 mm to 15.0 mm in thickness and with hardness of 10 IRHD to 35 IRHD. It is permissible to test the one with 6.0 mm or more thickness and with hardness of 10 IRHD to 35 IRHD.
 - Notes (2) The hardness values in 85 IRHD to 95 IRHD and 30 IRHD to 35 IRHD obtained by N method do not exactly coincide with the values by H method and L method, but the discrepancy does not come into technical problem, generally speaking.

(3) The testing apparatus for M method is the one prepared by miniaturizing the testing apparatus for N method by about one-sixth to measure the test piece with thin thickness, therefore the depth of plunger indentation by M method is just one-sixth that by N method. The results given by M method are not always coincident with the results given by N method because of the surface effect of rubber or slight roughness of the surface.

4.3 Testing apparatus

4.3.1 Outline of testing apparatus The testing apparatus is composed of a holding base for test piece by which a test piece is kept, an annular pressure foot by which the surface of a test piece is pressed, a plunger, with a ball-type lower end, set at the center of hole of pressure foot, a device for loading which gives an indenting force on a plunger to make an indentation on a test piece, a measuring device to measure depth of an indentation impressed on a test piece, and a vibrating device to lessen friction. The dimensions of main parts and the specification of force are shown in Table 2.

A thermostat may be provided for measuring a test temperature other than standard condition of laboratory.

Type Diameter of tests of ball of	Face of pressure foot			Force applying at ball of plunger end			
plunger end mm		Diameter mm	Diameter of hole mm	Force exerted on face of pressure foot	Contact force	Indenting force	Total
H method	1.00 ± 0.01	20 ± 1	6 ± 1				
N method	2.50 ± 0.01	20 ± 1	6 ± 1	$8.3 \pm 1.5 \text{ N}$ {846 ± 153 gf}	$0.30 \pm 0.02 \text{ N}$ $(30.6 \pm 2.0 \text{ gf})$	$5.40 \pm 0.01 \text{ N}$ $\{550.6 \pm 1.0 \text{ gf}\}$	$5.70 \pm 0.03 \text{ N}$ $\{581.2 \pm 3.1 \text{ gf}\}$
L method	5.00 ± 0.01	22 ± 1	10 ± 1				
M method	0.395 ± 0.005	3.35 ± 0.15	1.00 ± 0.15	(4) 235 ± 30 mN (24.0 ± 3.1 gf)	8.3 ± 0.5 mN {0.85 ± 0.05 gf}	$145 \pm 0.5 \text{ mN}$ { $14.79 \pm 0.05 \text{ gf}$ }	$153 \pm 1 \text{ mN}$ { $15.60 \pm 0.10 \text{ gf}$ }

Table 2 Main dimensions and forces of testing apparatus

Note (4) When in M method a pressure adjusting spring installed at the bottom of a test-piece holding base makes pressure adjustment, the pressure adjusting spring must be controlled to be $(380\pm30)\,\mathrm{mN}$ { $(38.7\pm3.1)\,\mathrm{gf}$ } because an indenting force 145 mN {14.8 gf} is added during measurement.

4.3.2 Face of pressure foot An annular pressure foot makes rectangular to a plunger. The diameter of face of pressure foot and the diameter of the hole for a plunger are as shown in Table 2. When the force exerted on the face of pressure foot is just as shown in Table 2, the pressure impressed on the surface of test piece becomes (30 ± 5) kPa $\{(0.306\pm0.051) \text{ kgf/cm}^2\}(5)$. In order to measure the relative displacement between the face of pressure foot (upper surface of test piece) and the plunger, the face of pressure foot shall be firmly united with the measuring device of the depth of indentation.

Note (5) Some combination of all tolerances shown in Table 2 does not always give nice coincidence with the description of pressure (30 ± 5) kPa $((0.306 \pm 0.051) \text{ kgf/cm}^2)$.

4.3.3 Plunger The plunger shall be vertical, and its lower end has spherical shape whose diameter shall be as shown in Table 2(6). The lower end ball of a plunger shall be kept a little upper than the face of pressure foot before contact force is applied.

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Note (6) The material of end ball shall be abrasion resistant and corrosion resistant.

When an end ball is connected with the body of plunger, the connected part must not be larger than diameter of the ball.

- **4.3.4 Loading device** Loading device shall accurately apply the contact force (7) and indenting force (8) specified in Table 2 to the end ball of a plunger.
 - Notes (7) Contact force means the force causing the end ball of a plunger to contact with surface of a test piece.
 - (8) Indenting force means the force to impress the end ball of a plunger into test piece after making contact.
- **4.3.5 Measuring device of indented depth** The measuring device for indented depth shall be capable of measuring indented depth of a plunger when indenting force is applied to a plunger, by which the indented depth or IRHD shall be directly read(9). The conversion from indented depth to IRHD can be done through Table 3, Table 4 and Table 5(10).
 - Notes (9) For the measuring device of indented depth, any of mechanical, optical, or electrical, is serviceable.
 - (10) Table 3 is for the conversion of H method, and Table 4 for N method. In case of M method, convert after making the indented depth shown in Table 4 one-sixth. Table 5 is the conversion table for L method.
- **4.3.6 Vibrating device** To overcome minute friction, it is preferable to install a vibrating device like an electric buzzer by which a testing apparatus is suitably vibrated. It can be eliminated if friction is completely removed.
- **4.3.7 Thermostat** The thermostat is needed when the test temperature other than standard condition of laboratory is employed for measuring hardness. The thermostat must keep the specified temperature in the tolerance of ± 2 °C. The annular foot with pressure face at lower end and a plunger shall penetrate through the upper part of the thermostat.

The part through which the plunger penetrates shall be made of the material with small thermal conductivity. The sensor for temperature measurement shall be installed at holding place of test piece or its vicinity, in the thermostat.

Table 3 Conversion table from indented depth (D) of a plunger to international rubber hardness degree (IRHD) (H method)

<i>D</i> mm	International rubber hardness degree IRHD	D mm	International rubber hardness degree IRHD	D mm	International rubber hardness degree IRHD
0.00	100.0	0.15	97.3	0.30	91.1
0.01	100.0	0.16	97.0	0.31	90.7
0.02	100.0	0.17	96.6	0.32	90.2
0.03	99.9	0.18	96.2	0.33	89.7
0.04	99.9	0.19	95.8	0.34	89.3
0.05	99.8	0.20	95.4	0.35	88.8
0.06	99.6	0.21	95.0	0.36	88.4
0.07	99.5	0.22	94.6	0.37	87.9
0.08	99.3	0.23	94.2	0.38	87.5
0.09	99.1	0.24	93.8	0.39	87.0
0.10	98.8	0.25	93.4	0.40	86.6
0.11	98.6	0.26	92.9	0.41	86.1
0.12	98.3	0.27	92.5	0.42	85.7
0.13	98.0	0.28	92.0	0.43	85.3
0.14	97.6	0.29	91.6	0.44	84.8

Table 4 Conversion table from indented depth (D) of a plunger to international rubber hardness degree (IRHD) (N method)

D	International rubber hardness	D mm	International rubber hardness degree IRHD	D mm	International rubber hardness degree IRHD	D mm	International rubber hardness degree IRHD
mm	degree IRHD						38.9
0.00	100.0	0.45	73.9	0.90	52.3	1.35	N .
0.01	100.0	0.46	73.3	0.91	52.0	1.36	38.7
0.02	99.9	0.47	72.7	0.92	51.6	1.37	38.4
0.03	99.8	0.48	72.2	0.93	51.2	1.38	38.2
0.04	99.6	0.49	71.6	0.94	50.9	1.39	38.0
0.05	99.3	0.50	71.0	0.95	50.5	1.40	37.8
0.06	99.0	0.51	70.4	0.96	50.2	1.41	37.5
0.07	98.6	0.52	69.8	0.97	49.8	1.42	37.3
0.08	98.1	0.53	69.3	0.98	49.5	1.43	37.1
0.09	97.7	0.54	68.7	0.99	49.1	1.44	36.9
0.10	97.1	0.55	68.2	1.00	48.8	1.45	36.7
0.11	96.5	0.56	67.6	1.01	48.5	1.46	36.5
0.12	95.9	0.57	67.1	1.02	48.1	1.47	36.2
0.12	95.3	0.58	66.6	1.03	47.8	1.48	36.0
0.13	94.7	0.59	66.0	1.04	47.5	1.49	35.8
0.15	04.0	0.60	65.5	1.05	47.1	1.50	35.6
0.15	94.0	0.60	t :	1.06	46.8	1.51	35.4
0.16	93.4	0.61	65.0		46.5	1.52	35.2
0.17	92.7	0.62	64.5	1.07		ŀ	35.0
0.18	92.0	0.63	64.0	1.08	46.2	1.53	
0.19	91.3	0.64	63.5	1.09	45.9	1.54	34.8
0.20	90.6	0.65	63.0	1.10	45.6	1.55	34.6
0.21	89.8	0.66	62.5	1.11	45.3	1.56	34.4
0.22	89.2	0.67	62.0	1.12	45.0	1.57	34.2
0.23	88.5	0.68	61.5	1.13	44.7	1.58	34.0
0.24	87.8	0.69	61.1	1.14	44.4	1.59	33.8
0.25	87.1	0.70	60.6	1.15	44.1	1.60	33.6
0.26	86.4	0.71	60.1	1.16	43.8	1.61	33.4
0.27	85.7	0.72	59.7	1.17	43.5	1.62	33.2
0.28	85.0	0.73	59.2	1.18	43.3	1.63	33.0
0.29	84.3	0.74	58.8	1.19	43.0	1.64	32.8
0.30	83.6	0.75	58.3	1.20	42.7	1.65	32.6
	82.9	0.75	57.9	1.21	42.5	1.66	32.4
0.31		H	57.5	1.22	42.2	1.67	32.3
0.32	82.2	0.77	1			1.68	32.1
0.33	81.5	0.78	57.0	1.23	41.9		31.9
0.34	80.9	0.79	56.6	1.24	41.7	1.69	31.9
0.35	80.2	0.80	56.2	1.25	41.4	1.70	31.7
0.36	79.5	0.81	55.8	1.26	41.1	1.71	31.6
0.37	78.9	0.82	55.4	1.27	40.9	1.72	31.4
0.38	78.2	0.83	55.0	1.28	40.6	1.73	31.2
0.39	77.6	0.84	54.6	1.29	40.4	1.74	31.1
0.40	77.0	0.85	54.2	1.30	40.1	1.75	30.9
0.41	76.4	0.86	53.8	1.31	39.9	1.76	30.7
0.42	75.8	0.87	53.4	1.32	39.6	1.77	30.5
0.42	75.2	0.88	53.0	1.33	39.4	1.78	30.4
0.43	74.5	0.89	52.7	1.34	39.1	1.79	30.2
0.44	17.0	0.03	""	1.01	55.*	1.80	30.0

Table 5 Conversion table from indented depth (D) of a plunger to international rubber hardness degree (IRHD) (L method)

D	International rubber hardness	D	International rubber hardness	D	International rubber hardness
mm	degree IRHD	mm	degree IRHD	mm	degree IRHD
1.10	34.9	1.80	21.3	2.50	14.1
1.12	34.4	1.82	21.1	2.52	14.0
1.14	33.9	1.84	20.8	2.54	13.8
1.16	33.4	1.86	20.6	2.56	13.7
1.18	32.9	1.88	20.3	2.58	13.5
1.20	32.4	1.90	20.1	2.60	13.4
1.22	31.9	1.92	19.8	2.62	13.3
1.24	31.4	1.94	19.6	2.64	13.1
1.26	30.9	1.96	19.4	2.66	13.0
1.28	30.4	1.98	19.2	2.68	12.8
1.30	30.0	2.00	18.9	2.70	12.7
1.32	29.6	2.02	18.7	2.72	12.6
1.34	.29.2	2.04	18.5	2.74	12.5
1.36	28.8	2.06	18.3	2.76	12.3
1.38	28.4	2.08	18.0	2.78	12.2
1.40	28.0	2.10	17.8	2.80	12.1
1.42	27.6	2.12	17.6	2.82	12.0
1.44	27.2	2.14	17.4	2.84	-11.8
1.46	26.8	2.16	17.2	2.86	11.7
1.48	26.4	2.18	17.0	2.88	11.6
1.50	26.1	2.20	16.8	2.90	11.5
1.52	25.7	2.22	16.6	2.92	11.4
1.54	25.4	2.24	16.4	2.94	11.3
1.56	25.0	2.26	16.2	2.96	11.2
1.58	24.7	2.28	16.0	2.98	11.1
1.60	24.4	2.30	15.8	3.00	11.0
1.62	24.1	2.32	15.6	3.02	10.9
1.64	23.8	2.34	15.4	3.04	10.8
1.66	23.5	2.36	15.3	3.06	10.6
1.68	23.1	2.38	15.1	3.08	10.5
1.70	22.8	2.40	14.9	3.10	10.4
1.72	22.5	2.42	14.8	3.12	10.3
1.74	22.2	2.44	14.6	3.14	10.2
1.76	21.9	2.46	14.4	3.16	10.1
1.78	21.6	2.48	14.3	3.18	9.9

4.4 Test piece

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4.4.1 Shape of test pieces Both surfaces of a test piece shall be smoothly flat and parallel each other(11). This test has been supposed to compare the test pieces having the same thickness.

Note (11) The surface such as unsmoothed, curved, or rough, does not give satisfactory results. For specially formed surface, however, such as rubber roll, this method can be applied.

The international rubber hardness testing method for curved test piece is shown in Informative reference.

4.4.2 Thickness

- (1) **H method and N method** The standard thickness of a test piece is 8.0 mm to 10.0 mm, but to get necessary thickness, it is permissible to pile smooth and parallel test pieces. Provided that the thickness of test pieces before piling shall be 2 mm or more, and 3 or more test pieces cannot be piled up. Even when nonstandard test piece other than above(12) is to be adopted, the thickness of the test piece must be 4.0 mm or more.
- (2) L method The standard thickness of a test piece is 10.0 mm to 15.0 mm, but to get necessary thickness, it is permissible to pile smooth and parallel test pieces. Provided that the thickness of test pieces before piling shall be 2 mm or more, and 3 or more test pieces cannot be piled up. Even when nonstandard test piece other than above(12) is to be adopted, the thickness of the test piece must be 6.0 mm or more.
- (3) **M method** The standard thickness of a test piece is (2.0 ± 0.5) mm. Even when nonstandard test piece other than above (12) is to be adopted, the thickness of the test piece must be 1.0 mm or more.
 - Note (12) The measured value resulted from nonstandard test piece, is not generally coincident with the measured value by standard test piece.

4.4.3 Lateral dimensions

(1) H method, N method, and L method The lateral dimension of a test piece shall be large enough to measure at the point which is apart from edge of the test piece by at least the distance shown in Table 6.

Table 6 Minimum distance of point for hardness measurement (point of end ball of plunger) from test-piece edge

	Unit: mm		
Thickness of a test piece	Minimum distance of point for hardness measurement from test-piece edge		
4.0	7.0		
6.0	8.0		
8.0	9.0		
10.0	10.0		
15.0	11.5		
25.0	13.0		

- (2) **M method** The lateral dimension of a test piece shall be large enough to measure at the point which is apart from edge of the test piece by at least 2.0 mm. When the test piece, with the thickness of 4.0 mm or more, which is not eligible for N method because of small lateral dimension or of not having large smooth area, is to be tested by M method, carry out test at the point apart from edge of the test piece as far as possible.
- 4.4.4 Sampling and preparation of test pieces The sampling and preparation of test pieces shall principally follow 6.5 of JIS K 6250.
- **4.4.5** Selection of test pieces The test pieces which contain alien matters, bubbles, or flaws shall not be used for tests.

4.5 Testing method

- 4.5.1 Testing conditions Testing conditions shall be as follows.
- (1) The standard conditions of a laboratory shall follow 6.1 of JIS K 6250.
- (2) Storing of sample and test pieces shall follow 6.2 of JIS K 6250.
- (3) The standard conditions of test pieces shall follow 6.3 of JIS K 6250.
- **4.5.2 Procedures** Sprinkle slightly talc on upper and back surfaces of a test piece to lessen friction between the end ball of a plunger and surface of a test piece. Place the test piece on the holding base of a test piece. Make the face of pressure foot touch with the surface of the test piece.
- (1) When the scale is graduated with IRHD, apply contact force to the plunger for 5 s, and adjust the scale to be 100. Then, apply indenting force for 30 s, and read directly hardness by IRHD.
- (2) When the scale is graduated with indented depth, apply contact force to the plunger for 5 s, and read the scale. Then, apply indenting force for 30 s, and read the scale. Calculate the difference between indentation by contact force and that by indenting force, and make this the indented depth D. Convert the value of D into IRHD making use of Table 3, Table 4, and Table 5.

While applying force, the slight vibration may be applied on the testing apparatus by a vibrating device to overcome the friction. Carry out measurements at 3 or 5 new points on a test piece at every measurement.

- 4.6 Arrangement of test results Round off the median of 3 or 5 measurements to whole number according to JIS Z 8401, and mark the sign IRHD after it. In case of standard hardness, after it mark "/" together with letter "S", and then mark "/" with sign as H, N, M, or L, which means testing method. In case of apparent hardness, after sign of IRHD mark "/" together with sign as H, N, M, or L, which means testing method.
 - Example 1 50 IRHD/S/N: means that standard test piece is measured by N method of international rubber hardness test, and standard hardness is 50 IRHD.

- Example 2 50 IRHD/M: means that nonstandard test piece is measured by M method of international rubber hardness test, and apparent hardness is 50 IRHD.
- 4.7 Record On test result, the following items shall be recorded.
- (1) Test result

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- (2) Shape and dimensions of test piece (whether standard test piece or nonstandard one; in case of nonstandard, whether curved surface or not; and in case of piled one, the number of piled pieces and its thickness)
- (3) Sampling and preparation methods of test pieces
- (4) Test temperature
- (5) Other items specially needed

5 Durometer hardness test

- **5.1 Purpose** This test shall be carried out to measure durometer hardness of vulcanized rubber.
- **5.2 Range of measurement** The measuring range of this test is decided according to the hardness of test piece at every testing method. The measuring range of each testing method is as follows.
- (1) **Type D durometer** The measuring range of type D durometer hardness is the range over A90 by type A durometer. When less than D20, measure by type A durometer.
- (2) **Type A durometer** The measuring range of type A durometer hardness is from A10 to A90, and when over A90, measure by type D durometer. When less than A20, measure by type E durometer.
- (3) **Type E durometer** The measuring range of type E durometer hardness is the range of less than A20 by type A durometer.

5.3 Testing apparatus

- **5.3.1 Outline of testing apparatus** The testing apparatus is composed of the face of pressure foot by which the surface of a test piece is pressed, indentor which protrudes from a central hole of face of pressure foot by action of a spring, and the graduation which indicates the distance (indenting depth) of indentor rejected by rubber cushion and which represents hardness itself.
- **5.3.2 Face of pressure foot** The face of pressure foot is perpendicular to the indentor, and its center has a hole for the indentor. The diameter of the hole, in case of type D and type A durometer, is $3.0^{+0.2}_{-0.5}$ mm, and in case of type E durometer, (5.4 ± 0.2) mm.

On the face of pressure foot, the distance from any place of its outer edge to the center of an indentor shall be, in case of type D and type A durometer, 6 mm or more, and in case of type E durometer, 7 mm or more.

5.3.3 Indentor The material of indentor shall be abrasion resistant and corrosion resistant, and it shall be accurately fixed at center of the hole of face of pressure foot. Its shape and dimensions are indicated in Fig. 1 for type D durometer, in Fig. 2 for type A durometer, and in Fig. 3 for type E durometer.

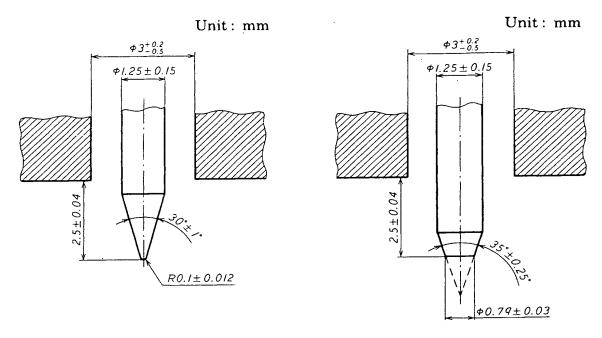


Fig. 1 Indentor for type D durometer

Fig. 2 Indentor for type A durometer

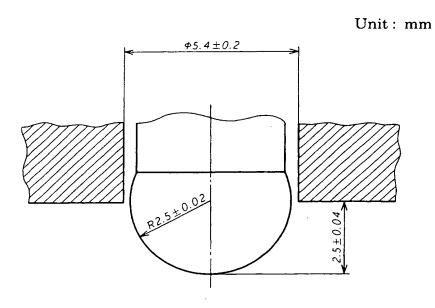


Fig. 3 Indentor for type E durometer

5.3.4 Scale When the scale indicates 0 (full protrusion), the point of the indentor shall protrude by (2.50 ± 0.04) mm beyond the face of the pressure foot.

When the scale indicates 100 (nil protrusion), the face of the pressure foot is in firm contact with a flat piece of glass, i.e. the point of the indentor shall be positioned on the same plane with the face of the pressure foot. The scale shall be graduated with equal intervals in the range between 0 to 100.

5.3.5 Spring There must be the following relation between the force of spring and the scale, that is, the durometer hardness.

(1) Type D durometer

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 $W_{\rm D} = 444.5 H_{\rm D} \left\{ w_{\rm D} = 45.33 H_{\rm D} \right\}$

where, W_D : force of spring of type D durometer (mN)

 w_D : force of spring of type D durometer $\{gf\}$

 H_D : hardness of type D durometer

(2) Type A and type E durometer

 $W_{\rm A} = 550 + 75H_{\rm A} \{ w_{\rm A} = 56.1 + 7.65H_{\rm A} \}$

where, W_A : force of spring of type A or type E durometer

(mN)

 w_A : force of spring of type A or type E durometer

{gf}

 H_A : hardness of type A or type E durometer

The tolerance of force shall be, in case of type D durometer, ±440 mN (±44.9 gf), and in case of type A and type E durometer, ±80 mN (±8.16 gf).

5.3.6 Calibration of spring Hold vertically the end point of indentor of a durometer on a balance not to give any interference between the balance and face of pressure foot, via a spacer (see Fig. 4). The cylindrical spacer with 2.5 mm height, in case of type D and type A durometer, measuring 1.25 mm in diameter, and in case of type E durometer, measuring 3 mm in diameter, has a wineglass shape where an indentor is to touch, in order to smoothly receive the end point of the indentor. Place a tare on the balance against the weight of the spacer. Place counterweight to get suitable scale, and confirm that the force (mN) shown here stays within the tolerance of specified force in 5.3.5. Carry out the above calibration using suitable scale interval.

The calibration of spring of a durometer may be done with an electrobalance other than chemical balance shown in Fig. 4. In this case, the measuring sensitivity of the force at end point of an indentor shall be, in case of type D durometer, 44 mN {4.5 gf} or less, and in case of type A and type E durometer, 8 mN {0.82 gf} or less.

The following method is permissible; place upside down the durometer, and directly apply the load on its indentor by counterweight. Provided that the correction about the mass of parts inside of the durometer shall be considered to prevent the discrepancy between this method and the method by Fig. 4. In this case, the accuracy on the mass of counterweight shall be ± 4.5 g or less in case of type D durometer and ± 0.82 g or less in case of type A and type E durometer.

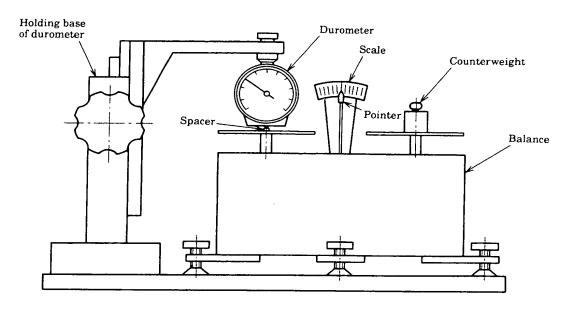


Fig. 4 Example of calibration apparatus of spring

5.4 Test piece

5.4.1 Shape and dimensions of test pieces The thickness of a test piece for type D and type A durometer is 6 mm or more. When it is less than 6 mm, pile them to make 6 mm or more for measurement. The thickness of a test piece for type E durometer is 10 mm or more, and in case of less than 10 mm, pile them to make 10 mm or more. The number of test pieces to pile shall be at most 3, and each of them shall have 2 mm or more thickness. The test result brought by piled up test piece doesn't generally coincide with the result by solid test piece(13). The lateral size of test piece shall be large enough to measure at the point where the end point of an indentor is apart 12 mm or more from the edge of the test piece.

Furthermore, the test piece shall have smooth surface spacious enough to make close contact with face of pressure foot of a durometer(14).

- Notes (13) To make comparison, it is necessary to use the test piece which has the same number for piling and the same thickness.
 - (14) The surface such as unsmoothed, curved, or rough, does not give satisfactory results. For specially formed surface, however, such as rubber roll, this method can be applied. In this case, the applicable limit of the durometer shall be definitely confirmed.
- 5.4.2 Sampling and preparation of test pieces The sampling and preparation of test pieces shall follow 6.5 of JIS K 6250.
- **5.4.3 Selection of test pieces** The test pieces which contain alien matters, bubbles, or flaws shall not be used for test.

5.5 Testing method

- **5.5.1 Testing conditions** Testing conditions shall be as follows.
- (1) The standard conditions of a laboratory shall follow 6.1 of JIS K 6250.

- (2) Storing of sample and test pieces shall follow 6.2 of JIS K 6250.
- (3) The standard conditions of test pieces shall follow 6.3 of JIS K 6250.
- 5.5.2 Procedures Place a test piece on a rigid, hard, and flat surface. Set a durometer so as to make an indentor rectangular to the target surface of a test piece. Contact closely as swiftly as possible the face of pressure foot with the target surface of the test piece without giving a impact, and read the scale within 1 s, to find the hardness of the test piece (15). But the agreement between the parties concerned with delivery may permit to read when a definite time passed after close contacting between them. The end point of the indentor of a durometer must be apart 12 mm or more from the edge of the test piece. Unless otherwise specified, the duration from close contacting to the finish of reading shall be recorded. The measuring points shall be 5, which are apart at least 6 mm each other, and carry out measurements 5 times on these points. When hardness shown by type A durometer is over A90, employ a type D durometer. When the hardness shown by type D durometer is less than D20, employ a type A durometer. If the hardness by type A durometer is less than A10, result is inaccurate, so don't record it.

When the hardness by a type A durometer is less A20, measure it with a type E durometer.

- Note (15) In order to get a good repeatability, the holding base for durometer may be used by which the durometer is vertically kept and target surface and indentor get right angle each other before measurement. In this case, it is recommended that the mass imposed on the pressing surface is 5.0 kg for type D durometer, and 1.0 kg for both type A and type E durometer.
- **5.6** Arrangement of test results Round off the median of 5 measurements to whole number according to **JIS Z 8401**, and mark sign D in case of type D durometer, sign A in case of type A durometer, and sign E in case of type E durometer, just before the rounded value. When the value was read when definite time passed after close contacting, mark sign "/" and then record the duration (s). When it is standard hardness, the above is followed by "/" and then by sign S.
 - Example 1 D85/15/S: means that standard test piece is measured by type D durometer hardness test, and the reading on standard hardness is 85 when 15 s passed after close contacting of face of pressure foot.
 - Example 2 A45/S: means that standard test piece is measured by type A durometer hardness test, and the reading on standard hardness is 45 within 1 s after close contacting of face of pressure foot.
 - Example 3 A45/15: means that nonstandard test piece is measured by type A durometer hardness test, and the reading on apparent hardness is 45 when 15 s passed after close contacting of face of pressure foot.
 - Example 4 E60: means that nonstandard test piece is measured by type E durometer hardness test, and the reading on apparent hardness is 60 within 1 s after close contacting of face of pressure foot.
- **5.7 Record** On test result, the following items shall be recorded.
- (1) Test result

(2) Shape and dimensions of test piece (whether standard test piece or nonstandard test piece; in case of piled up test piece, the number of piled pieces, and its thickness)

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- (3) Sampling and preparation methods of test pieces
- (4) Other items specially needed

6 IRHD pocket hardness test

6.1 Purpose This test shall be carried out to measure the international rubber hardness degree of vulcanized rubber by IRHD pocket hardness meter, and abbreviated P method.

6.2 Testing apparatus

- **6.2.1 Outline of testing apparatus** The testing apparatus is composed of a face of pressure foot to press the surface of a test piece, indentor which protrudes from a central hole of face of pressure foot by action of a spring, and a mechanism indicating the protruded length of the indentor.
- **6.2.2 Face of pressure foot** The face of pressure foot, measuring (20 ± 2.5) mm sided square, has a hole with 2.0 mm to 3.0 mm diameter at its center.
- **6.2.3 Indentor** The end of the indentor shall make a hemisphere with 1.55 mm to 1.60 mm diameter.
- **6.2.4** Indicating mechanism The indicating mechanism shows the protruded length of an indentor from face of pressure foot, and it shall have been calibrated to read directly the international rubber hardness degree by IRHD. When the longest protruded length of 1.65 mm is given, it must show 28 IRHD, and when the face of pressure foot is let contact with a flat glass, that is, no protruded, it must show 100 IRHD.
- **6.2.5** Spring Spring can apply constant force of (2.65 ± 0.15) N $\{(270.3 \pm 15.3)$ gf} to an indentor in the range from 28 IRHD to 100 IRHD.
- **6.2.6** Calibration of hardness meter IRHD pocket hardness meter shall be calibrated and adjusted using a standard rubber block whose international rubber hardness degree has been known. Only when the standard rubber block cannot be used, it is preferably calibrated with mechanical method.

Press the IRHD pocket hardness meter on a flat glass plate, and adjust the scale to get 100 IRHD. Making use of a set of standard rubber blocks from 30 IRHD to 90 IRHD, calibrate IRHD pocket hardness meter. The set of standard rubber blocks is stored in a container with a suitable cover after being sprinkled with talc powder, in order to prevent the influences by light, heat, oil, or grease. It consists of at least 6 test pieces. These standard blocks must be calibrated with the international rubber hardness test specified in 4 at intervals not exceeding six months. It is advisable that the IRHD pocket hardness meter, which is used daily, is calibrated at least once a week with standard rubber block.

Remarks: When IRHD pocket hardness meter is calibrated with mechanical method or adjusted, the instruction manual issued by the manufacturer shall be depended.

6.3 Test piece

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6.3.1 Shape and dimensions of test pieces The thickness of a test piece shall be 6 mm or more. When it is less than 6 mm, the test piece which was prepared by piling up to 6 mm or more can be used, but the number of piling up shall be 3 or less, and each of them shall have 2 mm or more thickness. The test result comes from piled test piece does not usually coincide with the test result by solid test piece (13). The lateral dimension of a test piece shall be large enough to measure at the point where the end point of an indentor is apart 12 mm or more from the edge of the test piece.

Test pieces shall have flat surface which is spacious to closely contact with the face of pressure foot of a hardness meter(16).

- Note (16) The surface such as unsmoothed, curved, or rough, does not give satisfactory results. For specially formed surface, however, such as rubber roll, this method can be applied. In this case, the applicable limit of the IRHD pocket hardness meter shall be definitely confirmed.
- **6.3.2 Sampling and preparation of test pieces** The sampling and preparation of test pieces shall follow **6.5** of **JIS K 6250**.
- **6.3.3 Selection of test pieces** The test pieces which contain alien matters, bubbles, or flaws shall not be used for test.

6.4 Testing method

- **6.4.1 Testing conditions** Testing conditions shall be as follows.
- (1) The standard conditions of a laboratory shall follow 6.1 of JIS K 6250.
- (2) Storing of sample and test pieces shall follow 6.2 of JIS K 6250.
- (3) The standard conditions of test pieces shall follow 6.3 of JIS K 6250.
- **6.4.2 Procedures** Place a test piece on a rigid, hard, and flat surface. Set an IRHD pocket hardness meter so as to make an indentor rectangular to the target surface of a test piece. Contact closely as swiftly as possible the face of pressure foot with the target surface of the test piece without giving a impact, and read the scale within 1 s, to find the hardness of the test piece. The end point of the indentor of an IRHD pocket hardness meter must be apart 12 mm or more from the edge of the test piece. Unless otherwise specified, read the value within 1 s after close contacting, but if the reading after special duration is specified, follow that specification. In this case, the duration from close contacting to the finish of reading shall be recorded. The measuring points shall be 5, which are apart at least 6 mm each other, and carry out measurements 5 times on these points.
- 6.5 Arrangement of test results Round off the median of 5 measurements to whole number according to JIS Z 8401, then mark sign IRHD after the value, and in case of standard hardness, after the value mark sign "/", then sign S, then again sign "/" and last sign P which means testing method. In case of apparent hardness, mark sign "/" after sign IRHD, then mark sign P which means testing method.

- Example 1 50 IRHD/S/P: means that standard test piece is measured by IRHD pocket hardness meter, and the standard hardness is 50 IRHD.
- Example 2 50 IRHD/P: means that nonstandard test piece is measured by IRHD pocket hardness meter, and the apparent hardness is 50 IRHD.
- 6.6 Record On test result, the following items shall be recorded.
- (1) Test result
- (2) Shape and dimensions of test piece (whether standard test piece or nonstandard test piece; in case of piled up test piece, the number of piled pieces, and its thickness)
- (3) Sampling and preparation methods of test pieces
- (4) Other items specially needed

ISO 7267/1:1986 Rubber-covered rollers—Determination of apparent hardness—

Part 1: IRHD method

ISO 7267/2: 1986 Rubber-covered rollers—Determination of apparent hardness—

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Part 2: Shore-type durometer method

Informative reference

International rubber hardness testing method for curved test piece

Introduction This Informative reference states the international rubber hardness testing method for curved test piece, and does not make a part of Standard.

1 Purpose This test shall be carried out to measure international rubber hardness degree of a test piece of vulcanized rubber whose target surface makes a curved surface. The measured values obtained by this method are always treated as an apparent hardness.

Remarks: The standards cited in this Informative reference are listed as follows.

ISO 48:1994 Rubber, vulcanized or thermoplastic—Determination of hardness (hardness between 10 IRHD and 100 IRHD)

ISO 7267/1: 1986 Rubber-covered rollers—Determination of apparent hardness—Part 1: IRHD method

ISO 7267/2: 1986 Rubber-covered rollers—Determination of apparent hardness—Part 2: Shore-type durometer method

2 Type of testing method

- (1) CH method (normal size curved surface test for high hardness)
- (2) CN method (normal size curved surface test for normal hardness)
- (3) CM method (microsize curved surface test for normal hardness)
- (4) CL method (normal size curved surface test for low hardness)
- 3 Scope CH method, CN method, CM method, and CL method are the modified H method, N method, M method, and L method for the purpose of making them applicable to the test piece whose target surface is curved, and there are the following two cases(1).
- (1) Test piece or sample is large enough to place the hardness testing apparatus on it.
- (2) Test piece or sample is so small that it must be placed on a holding base together with a hardness testing apparatus. The case where the sample is put on a flat sample base which makes one body with a testing apparatus, is included in this case.
 - Note (1) Generally, these tests are carried out directly on products, so that the thickness of rubber is not constant, and in many cases, the lateral distance from the end ball of a plunger to the edge of sample is smaller than the smallest distance shown in 4.4.3 in the body of this Standard, and the influence owing to the distance from the edge is not negligible.

Therefore, the measured values resulted from these methods don't coincide with the values obtained by the measurements of the plate-type test pieces with flat parallel surfaces and the same thickness as that of standard test pieces or products which are specified in H method, N method, M method and L method.

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This means that, the results obtained by measuring curved surface are the peculiar measurements which are applicable only to the test pieces or the products having special shape and special dimensions and further being kept in special method. In extreme case, these measured values show discrepancy of 10 IRHD from the standard hardness. The measured values on the surface buffed to eliminate covered cloth or treated specially, shows a little difference value from the value on flat surface which has been finished with molding.

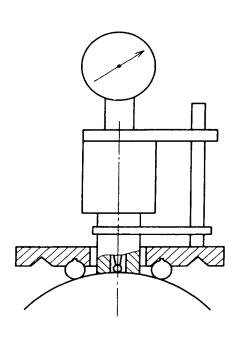
4 Testing apparatus

- **4.1 General matters** Basically, testing apparatus follows **4.3** of the body of this Standard, but the following gives difference.
- **4.2 Testing apparatus for cylindrical surface of 50 mm or more radius** As shown in Informative reference Fig. 1, the bottom base of the testing apparatus has a hole through which annular pressure foot can penetrate, for the measurement even when sample is put under the base.

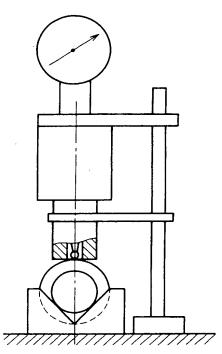
There are two cylindrical surfaces which are parallel each other under the base, and these are parallel to the horizontal surface of the base. The diameter of these cylinders and the distance between them shall be suitable for setting up testing apparatus on the target curved surface of sample. Alternatively, the base, on which adjustable legs with universal joints are attached to comply with the target curved surface, may be used.

- 4.3 Testing apparatus for two-way curved surface of 50 mm or more radius. The testing apparatus with adjustable legs with universal joints shown in 4.2 can be used.
- 4.4 Testing apparatus for cylindrical surface and two-way curved surface of 4 mm to 50 mm radius When target surface is too small to set a testing apparatus on it, as shown in Informative reference Fig. 2, fix test piece or sample using a special jig, V-block, or the like, and set the plunger to be perpendicular onto the target surface. When a small test piece is fixed on a sample table, wax may be used(2)(3).
 - Notes (2) The testing apparatus for M method shall be generally used only for the test piece whose thickness is 4 mm or less.
 - (3) The testing apparatus for M method, whose sample table is forced up owing to the action of a spring, is not suitable for the large-sized test piece or sample having curved surface with large radius.

4.5 Testing apparatus for small type O-ring and curved sample of 4 mm or less radius In these cases, hold a test piece on the table of testing apparatus using a suitable jig, block, wax, or the like. Carry out measurement using a testing apparatus of M method. The test piece having the minimum radius of 0.8 mm or less cannot be measured.



Informative reference Fig. 1 Example of setting a testing apparatus for sample with large diameter



Informative reference Fig. 2
Example of setting a testing apparatus for sample with small diameter

5 Test pieces

- 5.1 General matters The test pieces for CH method, CN method, CM method, and CL method are the products or the pieces prepared by cutting the products. The bottom side of the test piece which has been cut out shall be held with suitable method. In case of the target surface is covered with cloth, it must be buffed before testing. In order to recover it from the influence by buffing, allow it to stand for 16 h or more under standard condition of laboratory, and then carry out conditioning under standard condition according to (3) of 4.5.1 in the body of this Standard. This duration may be included in the duration for recovering.
- 5.2 Sampling and preparation of test pieces The sampling and preparation of test pieces shall follow 4.4.4 in the body of this Standard.
- **5.3** Selection of test pieces The selection of test pieces shall follow **4.4.5** in the body of this Standard.
- 6 Testing method The testing method shall follow 4.5 in the body of this Standard.

7 Arrangement of test results Round off the median of 3 or 5 measurements to whole number according to **JIS Z 8401**, and then mark sign IRHD after the value. After that, mark sign "/", and then mark CH, CN, CM, or CL which means testing method.

Example: 50 IRHD/CM: means that a curved test piece is measured by CM method of international rubber hardness curved-surface test, and the hardness is 50 IRHD.

- 8 Record On test result, the following items shall be recorded.
- (1) Test result
- (2) Shape and dimensions of test pieces
- (3) Sampling and preparation methods of test piece
- (4) Test temperature
- (5) Other items specially needed



Translated and Published by Japanese Standards Association

JIS K 6262: 1997

Permanent set testing methods for rubber, vulcanized or thermoplastic

ICS 83.060

Descriptors: vulcanized rubber, vulcanized materials, permanent, tension set, strain measurement, strain, mechanical behaviour of mateirals

Reference number: JIS K 6262: 1997 (E)

Permanent set testing methods for rubber, vulcanized or thermoplastic

JIS K 6262 : 1997

Introduction This Japanese Industrial Standard has been prepared on the basis of, the 2nd edition of ISO 815, Rubber, vulcanized or thermoplastic — Determination of compression set at ambient, elevated or low temperatures published in 1991, and ISO/DIS 2285, Rubber, vulcanized or thermoplastic — Determination of tension set at normal and high temperatures published in 1996, without any modification in technical content.

1 Scope This Japanese Industrial Standard specifies the testing methods to measure tension set and compression set of vulcanized rubber and thermoplastic rubber (hereafter referred to as "vulcanized rubber").

Remarks 1 The standards cited in this Standard are listed as follows:

JIS B 0601 Surface roughness - Definitions and designation

JIS K 6200 Glossary of terms used in rubber industry

JIS K 6250 General rules of physical testing methods for rubber, vulcanized or thermoplastic

JIS K 6251 Tensile testing methods for vulcanized rubber

JIS K 6257 Accelerated aging test methods for vulcanized rubber

JIS Z 8401 Rules for rounding off of numerical values

2 The International Standards corresponding to this Standard are listed as follows:

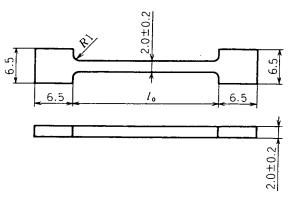
ISO 815:1991 Rubber, vulcanized or thermoplastic — Determination of compression set at ambient, elevated or low temperatures

ISO/DIS 2285: 1996 Rubber, vulcanized or thermoplastic — Determination of tension set at normal and high temperatures

- 2 Definitions For the purposes of this Standard, the definitions given in JIS K 6200 and JIS K 6250 apply.
- 3 Type of tests Permanent set testing methods are classified into the following two types.
- (1) Tension set test
- (2) Compression set test
- 4 Testing method of tension set
- 4.1 Purpose This test is carried out to measure the permanent set caused by tension applied to vulcanized rubber which is used for the parts under static tension.

Unit: mm

Unit: mm



 l_0 means 25.0 mm to 50.0 mm.

Fig. 1 Shape and dimensions of letter "I"-type test piece

(3) Ring-type test piece The shape and dimensions of ring-type test piece are shown in Table 1.

Table 1 Shape and dimensions of ring-type test piece

Shape Dimensions of main part

Outside diameter Inside diameter Thickness

Large-sized test piece 52.6 ± 0.2 44.6 ± 0.2 4.0 ± 0.2 Small-sized test piece 33.5 ± 0.2 29.5 ± 0.2 2.0 ± 0.2

- 4.3.2 Sampling and preparation of test pieces The sampling and preparation of test pieces shall follow 6.5 of JIS K 6250.
- 4.3.3 Number of test pieces Number of test pieces shall be 3 pieces.
- 4.3.4 Measurement of thickness and width of test piece The measurement of thickness and width of a test piece shall follow 6.6 of JIS K 6250.
- 4.3.5 Length of measured part of test piece
- (1) Strip-type test piece On the strip-type test piece, bench marks for elongation measurement are accurately and clearly marked between its parallel part with making the center of bench marks the center of test piece. The length for measurement shall be the gauge length.
- (2) Letter "I"-type test piece In case of letter "I"-type test piece, the length of narrowed part is made the length for measurement i.e. the guage length, instead of marking bench marks.

(7) The rate by which tensile procedure or compressive procedure is carried shall be 2 mm to 10 mm per second.

4.4.2 Procedures Procedures shall be carried out as follows.

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- (1) Measurement of distance between bench marks The distance between bench marks shall be measured at standard condition of a laboratory according to 4.3.5 to the nearest 0.1 mm.
- (2) Way to apply strain Under standard condition of a laboratory, mount a test piece on a straining device, elongate it up to the length corresponding to specified tensile strain with the rate of 2 mm to 10 mm per second, and keep it as it is. In this time, be careful not to give over elongation than that given by specified tensile strain. In case of a ring-type test piece, rotate the pulley by hands to make the strain uniform.

When bench marks for elongation measurement are to be put on a ring-type test piece, try the gauge length to fall in the center of the pulley. When 10 minutes to 20 minutes passed after specified tensile strain was got, measure again the length between bench marks, confirm if it falls in the range of tolerance. In case of using a inside diameter of a ring-type test piece, calculate it using diameter of a pulley and distance between pulleys, and measure it to the nearest 0.1 mm. If it is out of the tolerance, discard that test piece, and retry with new test piece.

- (3) **Heat treatment** During from 20 minutes to 30 minutes after specified tensile strain is given to the test piece, place it in a thermostat which has been kept at specified test temperature. The test shall start at the point when 30 minutes passed after giving tensile strain to the test piece. Heat it at test temperature for specified duration.
- (4) Measurement of elongation after heat treatment After heat treatment was finished, draw out the straining device from the thermostat, immediately let it shrink at the rate of 2 mm to 10 mm per second, take out the test piece from the straining device, place it on a wooden plate, and allow it to stand under standard condition of a laboratory. After 30⁺³ minutes, measure the distance between bench marks of the test piece to the nearest 0.1 mm(⁴).
 - Note (4) Alternative method is as follows. After heat treatment was finished, draw out the straining device from the thermostat, and allow the whole straining device to stand under standard condition of laboratory for 30 minutes to 35 minutes. After that, let the tension shrink at the rate of 2 mm to 10 mm per second, take out the test piece from the straining device, place it on a wooden plate, and measure the distance between bench marks of the test piece 30⁺³ minutes later. When this method is used, mention this on record.
- **4.5** Calculation Calculate tension set according to the following formula (2).

$$T_{\rm S} = \frac{l_2 - l_0}{l_1 - l_0} \times 100 \ ... \tag{2}$$

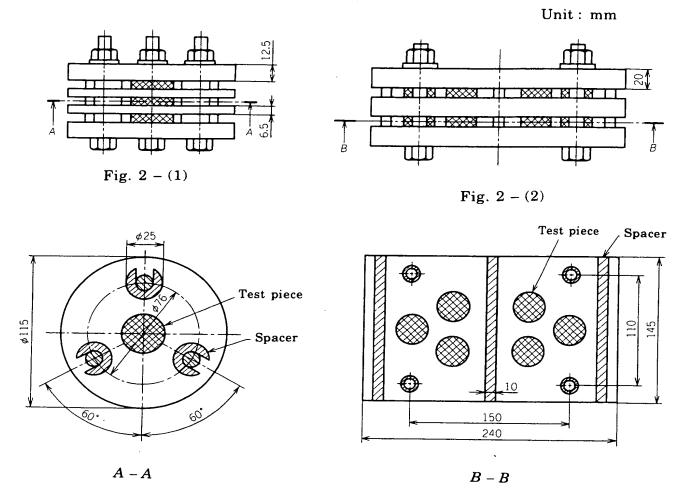


Fig. 2 Example of compression apparatus

- (1) Compression plates Compression plates shall be a chromium plated steel plate whose surface is in parallel and finished smoothly enough or polished stainless steel plate and be 0.4 μmRa or less(5), and comply with the following conditions.
 - (a) With sufficient rigidity, and making no deformation due to the stress given by a test piece.
 - (b) With sufficient size in which compressed test piece can surely stay in the surface of the compression plates.
 - (c) When a test piece is compressed, they shall be capable of keeping complete parallel condition.
 - Note (5) This means the average at central line. See JIS B 0601.
- (2) **Spacer** Spacer, made of mild steel, has a size not to touch with a test piece while compression is carried out. Thickness of a spacer depends on the compression percentage by which a test piece is compressed. Table 2 shows the thickness of a spacer when the compression percentage, such as 25 %, 15 %, or 10 %, is employed. The thickness of each of spacers which are used as a set for one test must be within ±0.01 mm of the average thickness of spacers in the set.

- Remarks 1 It is desirable to use the test pieces prepared through mold cure. Otherwise, they are prepared by cutting out product, or by punching disks from sheet according to the dimensions specified in 5.3.1 and then piling up them. In this case, number of disks piling up shall be 3 or less.
 - 2 When making piling up, never use adhesives. Pile up disks, and compress them for one minute to make a few percent compression, to get a close adhesion. Then, the total thickness of the piling up must be measured.
 - The result from the piled-up test piece and that from a solid test piece do not give the same result, therefore it is advisable to avoid direct comparison between them.
- 5.3.3 Number of test pieces The number of test piece shall be 3 pieces.
- 5.3.4 Measurement of thickness of test piece The measurement of thickness of test piece shall follow A method in (1.1) of 6.6.1 of JIS K 6250, under the standard condition of laboratory. The diameter of a probe shall be 4 mm to 10 mm. Measure the thickness of a test piece to the nearest 0.01 mm.
- 5.3.5 Selection of test pieces The test piece which contains alien matters, bubbles, or flaws shall not be used for test.

5.4 Testing method

- 5.4.1 Test conditions Test conditions shall be as follows.
- (1) The standard conditions of laboratory shall follow 6.1 of JIS K 6250.
- Storing of sample and test pieces shall follow 6.2 of JIS K 6250.
- The standard conditions of test pieces shall follow 6.3 of JIS K 6250.
- (4) Unless otherwise specified, the test temperature shall be selected among the following.

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(23\pm2) °C, (40\pm1) °C, (55\pm1) °C, (70\pm1) °C, (85\pm1) °C, (100\pm1) °C,
(125\pm2)~^{\circ}\mathrm{C},~(150\pm2)~^{\circ}\mathrm{C},~(175\pm2)~^{\circ}\mathrm{C},~(200\pm2)~^{\circ}\mathrm{C},~(225\pm2)~^{\circ}\mathrm{C},~(250\pm2)~^{\circ}\mathrm{C}
```

- The duration for test shall be any of 24_{-2}^{0} hours, 72_{-2}^{0} hours, 168_{-2}^{0} hours, and multiple 168 hours.
- (6) The percentage by which test piece is compressed shall be as shown in Table 4 depending on the hardness of the test piece.

- (6) Confirmation of test piece after test Halve the tested test piece along diameter line, if such inside faults as bubbles are found, don't adopt this test result.
- 5.5 Calculation Calculate compression set according to the following formula (3).

$$C_{\rm S} = \frac{t_0 - t_2}{t_0 - t_1} \times 100 \dots (3)$$

where,

 $C_{\rm S}$: compression set (%)

t₀: original thickness of test piece (mm)

 t_1 : thickness of spacer (mm)

 t_2 : thickness of test piece 30 minutes later from being let free from compression apparatus (mm)

- 5.6 Arrangement of test result Principally, test result shall be expressed with the average of 3 values obtained by the measurement of 3 test pieces, and the averages shall be rounded off according to JIS Z 8401 to whole number. If each measured value of three test pieces is dispersed by larger value than the one-tenth (absolute value) of average or the 2 % (absolute value) of its median whichever is the larger, take 3 more test pieces, repeat the test, and express with the median of totalled 6 values.
- 5.7 Record On test result, the following items shall be recorded.
- (1) Compression set
- (2) Compression percentage applied on test piece
- (3) Shape and dimensions of test piece
- (4) Sampling and preparation methods of test pieces (in case of piling up, the number of its piled pieces)
- (5) Number of test pieces
- (6) Duration of test and test temperature
- (7) Whether used or not lubricant
- (8) Other items specially needed

Errata for JIS (English edition) are printed in *Standardization Journal*, published monthly by the Japanese Standards Association, and also provided to subscribers of JIS (English edition) in *Monthly Information*.

Errata will be provided upon request, please contact:

Standardization Promotion Department, Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN
TEL. 03-3583-8002 FAX. 03-3583-0462